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WATER METALS NO. 4, STUDY NUMBER 30. REPORT OF A STUDY CONDUCTED BY ANALYTICAL REFERENCE SERVICE

Earl F. McFarren, et al

Public Health Service
Cincinnati, Ohio

1968

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ENVIRONMENTAL HEALTH SERIES
Urban and Industrial Health

WATER METALS NO. 4

STUDY NUMBER 30

ANALYTICAL REFERENCE SERVICE

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U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service

WATER METALS NO. 4

Study Number 30

Report of a Study Conducted by

ANALYTICAL REFERENCE SERVICE

Earl F. McFarren, John H. Parker, and
Raymond J. Lishka
TRAINING PROGRAM
National Center for Urban and Industrial Health

U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Bureau of Disease Prevention and Environmental Control
Cincinnati, Ohio
1968

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Public Health Service Publication No. 999-UIH-8

FOREWORD

The Analytical Reference Service (ARS) is conducted by the Training Program of the National Center for Urban and Industrial Health to evaluate laboratory methods in the environmental field. Cooperative studies by member organizations, who analyze identical samples and critically review methodology, provide the mechanism for:

> Evaluation of analytical procedures, including precision and accuracy, by comparison of the procedures and results reported by participating laboratories.
>
> Exchange of information regarding method characteristics.
>
> Improvement or replacement of existing methods by development of more accurate procedures, and development of new methodology for determination of new pollutional compounds.

Samples are designed to contain measured amounts of selected constituents. Decisions as to qualitative makeup are made by the ARS staff, the membership, and consultants. Notice of each study is sent to the entire membership. To those who desire to participate, a portion of the study sample is sent, along with data forms for reporting numerical values, a critique of the procedures used, comments on modifications, sources of error, difficulties encountered, or other pertinent factors. The results and comments received are compiled, and a report of each study is prepared.

Initially directed toward examination of water, studies now include air, milk and food. Some studies are periodically repeated for the advantage of new members, to evaluate new methods, or to reevaluate existing methods.

The selection of studies is guided by the responses to questionnaires periodically circulated among the membership, which now includes over 250 Federal, state, and municipal agencies; industries; universities; consulting firms; and foreign agencies.

James P. Sheehy, Chief
Training Program

ACKNOWLEDGEMENTS

Michael J. Taras, Chairman, Joint Editorial Board of Standard Methods for the Examination of Water and Wastewater suggested this study in order to obtain information on the usefulness of atomic absorption spectrophotometry in the analysis of water metals.

Dr. G. K. Murthy, Research Chemist, Environmental Sanitation Program, National Center for Urban and Industrial Health, and Mr. D. G. Ballinger, Assistant Chief for Laboratories, Division of Pollution Surveillance, Federal Water Pollution Control Administration, reviewed and suggested some changes in the method proposed for collaborative study. Many of these changes were adopted and formed a part of the method which was used by the participants in this study.

Mr. J. M. Matthews, Chemist, Analytical Reference Service, prepared the samples and checked the accuracy and stability of the samples by performing many analyses of aliquots of the samples both before and after distribution.

STUDIES COMPLETED AND REPORTED

Water-Minerals	Calcium, magnesium, hardness, sulfate, chloride, alkalinity, nitrite, nitrate, sodium, and potassium. Studies completed in 1956, 1958, and 1961.
Water-Metals	Lead, copper, cadmium, aluminum, chromium, iron, manganese, and zinc. Studies completed in 1957 and 1962. These same metals plus silver. Study completed in 1965. All of these same metals, except for the substitution of magnesium instead of aluminum, analyzed by atomic absorption in 1967.
Water-Fluoride	Fluoride in the presence and absence of interferences, with and without distillation by a specified procedure. Studies completed in 1958 and 1961.
Water-Radioactivity	Gross beta activity. Studies completed in 1959 and 1961. Gross beta and strontium-90 activity. Study completed in 1963.
Water-Surfactant	Surfactant in various waters. Studies completed in 1959 and 1963.
Water-Oxygen Demand	Biochemical oxygen demand and chemical oxygen demand. Study completed in 1960. Chemical oxygen demand. Study completed in 1965.
Water-Trace Elements	Arsenic, boron, selenium, and beryllium. Study completed in 1962. These same metals plus vanadium. Study completed in 1966.
Freshwater Plankton	Evaluation of the precision and accuracy obtainable by the use of various methods of plankton counting and identification. Study completed in 1964.
Water-Nutrients	Silicate, phosphate, ammonia nitrogen, organic nitrogen, and nitrate nitrogen. Study completed in 1966.

Water-Phenols	Phenol and 2,4-dichlorophenol in water by two specified methods. Study completed in 1966.
Water-Cyanides	Potassium cyanide and potassium ferricyanide in water by two specified methods. Study completed in 1967.
Air-Inorganics	Chloride, sulfate, fluoride, and nitrate in aqueous solution and on glass-fiber, high-volume filter mats. Study completed in 1958.
Air-Lead	Filter paper tape impregnated with lead. Study completed in 1961.
Air-Particulates	Microscopic identification of some common atmospheric particulates. Study completed in 1964.
Air-Sulfur Dioxide	Sulfur dioxide in air by a specified method. Study completed in 1963.
Water-Pesticides	Lindane, heptachlor epoxide, DDE, and dieldrin in water. Study completed in 1965.
Food-Pesticides	DDT in milk. Study completed in 1962. Lindane, heptachlor epoxide, DDE, and dieldrin in food. Study completed in 1965.

PARTICIPANTS IN THIS STUDY

Armco Steel Corporation, Middletown, Ohio
Calgon Corporation, Pittsburgh, Pennsylvania
Carnation Research Laboratories, Van Nuys, California
Central Electricity Research Laboratories, Surrey, England
Connecticut State Department of Health, Hartford
Cyrus Wm. Rice and Company, Pittsburgh, Pennsylvania
DHEW, PHS, Food Chemistry, Cincinnati, Ohio
DHEW, PHS, Water Supply, Cincinnati, Ohio
Durham Department of Water Resources, North Carolina
East Bay Municipal Utility District, Oakland, California
Emery Industries, Incorporated, Cincinnati, Ohio
Fresno County Health Department, California
Goodyear Atomic Corporation, Piketon, Ohio
Idaho Department of Health, Boise
Illinois State Water Survey, Urbana
Indiana State Board of Health, Indianapolis
Institute for Vattenoch Luftvardsforskning, Stockholm, Sweden
Iowa State Hygienic Laboratory, Des Moines
Kennecott Copper Corporation, Salt Lake City, Utah
Lawrence Experiment Station, Massachusetts
Louisiana State Board of Health, New Orleans
Metropolitan Sanitary District of Greater Chicago, Illinois
Minnesota Department of Health, Minneapolis
Nalco Chemical Company, Chicago, Illinois
New Hampshire Water Pollution Commission, Concord
New Jersey State Department of Health, Trenton
New York Department of Health, New York
New York Department of Water Supply, Gas and Electricity, Shokan
New York University Medical Center, New York
North Carolina Department of Water Resources, Raleigh
Ohio State Department of Health, Columbus
Ontario Water Resources Commission, Canada
Perkin-Elmer Corporation, Norwalk, Connecticut
Philadelphia Department of Public Health, Pennsylvania
Regional Environmental Health Laboratory (SGHK), Kelly AFB, Texas
Reynolds Electrical and Engineering Company, Inc., Las Vegas, Nevada
Rutgers - The State University, New Brunswick, New Jersey
Sandia Corporation, Albuquerque, New Mexico
South Carolina State Board of Health, Columbia
Tennessee Valley Authority, Chattanooga
United Kingdom Atomic Energy Authority, Didcot, Berks, England
U.S. Army Environmental Hygiene Agency, Edgewood Arsenal, Maryland
USDA, Soils Laboratory, Beltsville, Maryland
USDI, Bureau of Reclamation, Denver, Colorado
USDI, FWPCA, Alaska Water Laboratory, College

USDI, FWPCA, Analytical Applications, Cincinnati, Ohio
USDI, FWPCA, Hudson Champlain Project, Metuchen, New Jersey
USDI, FWPCA, Robert S. Kerr Water Research Center, Ada, Oklahoma
USDI, Geological Survey, Denver, Colorado
USDI, Geological Survey, Little Rock, Arkansas
USDI, Geological Survey, Sacramento, California
U.S. Industrial Chemical Company, Tuscola, Illinois
University of Vermont, Burlington
Utah State Department of Health, Salt Lake City
Virginia State Water Control Board, Richmond
W. R. Grace and Company, Lake Zurich, Illinois
Water Service Laboratories, Incorporated, New York, New York
York Research Corporation, Stamford, Connecticut

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ABSTRACT

In this study, three samples containing between 0.005 and 5.0 mg per liter of each of nine metals – zinc, chromium, copper, magnesium, manganese, silver, lead, cadmium, and iron – were provided. Each participant was requested to do a single analysis for each of the metals in each of the three samples by the provided atomic absorption spectrophotometric method. This method, depending upon the sensitivity of the instrument (burner, tube, etc.) available, gave the participant a choice of aspirating the sample directly into the flame or of chelating with ammonium pyrrolidine dithiocarbamate and extracting into methyl isobutyl ketone before aspirating.

The results obtained were evaluated in terms of whether the sensitivity of the method was sufficient to permit the measurement of the metal with a reasonable degree of precision and accuracy at the concentration prescribed by the drinking water standards.

In the determination of zinc, copper, magnesium, and iron, the procedure of aspirating the sample directly into the flame was found to meet the established criteria regardless of which type of burner or instrument was used. The same procedure was also found acceptable for the determination of chromium, manganese, and silver, provided a Boling burner was used. Acceptable results were obtained for lead only by chelation and extraction of the sample before aspiration. In the determination of cadmium, neither procedure was acceptable.

WATER METALS NO.4

PURPOSE OF THE STUDY

The few laboratories that analyzed the previous "Water Metals No. 3" study samples by atomic absorption spectrophotometry obtained favorable results. This study was, therefore, designed specifically to evaluate this new instrumental technique.

In particular, it seemed desirable to determine (1) whether the methods that have been developed for determining the concentration of metals by atomic absorption spectrophotometry in natural and treated waters in the absence of gross pollution are sufficiently sensitive to measure the quantities specified by established standards, (2) the precision and accuracy of this technique, and (3) the suitability of the method for inclusion as a "Standard Method" of water analysis for these metals.

DESIGN OF THE STUDY

To reduce shipping costs, the samples were prepared as concentrated solutions and shipped in sterile, sealed, 25-ml glass ampoules. Three samples were prepared and each participant was requested to do a single analysis by atomic absorption spectrophotometry for each of nine metals in each of the three samples.

The concentrations of the metals in the samples were such that when the participant diluted 5 ml of each sample to 1 liter with distilled water, as instructed, the samples would contain (as indicated in Table 1) concentrations of the metals approximately in the range of the established drinking water standards.

To obtain greater stability and prevent precipitation or adsorption of the metals on walls of the container, the pure metal or its salt was dissolved in nitric acid and excess acid added to reduce the pH of the final mixture to about pH 1.0

In analyzing the samples, the participants were instructed to use the procedures sent with the sample (see appendix A) and no other.

TREATMENT OF THE DATA

As the results of analysis were received from each participant, the data were reviewed, and in the case of a suspected mathematical error or misplaced decimal point, the reporting laboratory was asked to check the calculations and to submit a corrected value if an error was found.

Table 1. Composition of Samples

Compound	Metal	Metal in diluted sample, mg/l		
		Sample 1	Sample 2	Sample 3
Zinc metal	Zn	0.05	0.50	1.00
$K_2Cr_2O_7$	Cr	0.20	0.10	0.05
Copper metal	Cu	0.05	0.25	1.00
$MgSO_4 \cdot 7H_2O$	Mg	0.20	0.10	0.05
$KMnO_4$	Mn	0.05	0.50	0.25
$AgNO_3$	Ag	0.05	0.10	0.20
$Pb(NO_3)_2$	Pb	0.20	0.10	0.05
Cadmium metal	Cd	0.01	0.05	0.10
$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	Fe	0.10	0.50	0.30

The data were then coded, key-punched on cards, and analyzed by electronic computer for normality of distribution and subsequent rejection of outliers that were nonrepresentative because of errors in calculation, dilution, or other indeterminate factors. The rejection of outliers was performed separately on the results obtained by different makes of instruments and types of burners, and again collectively, regardless of instrument but separately according to whether the samples were concentrated by chelating and extraction, or simply aspirated directly into the flame.

The computer was also programmed to plot the data on probability paper in order to verify the above more objective technique for rejection of outliers. After rejection of outliers, the data were then statistically analyzed by computer for precision and accuracy.

Bar graphs are included in the text to provide a pictorial display of the data. Rejected values are indicated by broken bars, and values are given for bars off the scale of the chart.

Table 2 lists the types and numbers of burner and instrument combinations used by the participants in this study. As can be seen, a great variety of instrument and burner combinations were used, and, hence, many of the results represent a single determination by that particular instrument and/or burner combination. As a result, it was not possible to make any statistically significant comparison of the types (manufacturers) of instruments used in this study. Therefore, in the summary tables in the discussion section of this report, those results representing the summation of only one or two determinations are not presented.

Table 2. Types and Numbers of Instrument and Burner Combinations Used in This Study

Instrument or Method No.	Manufacturer	Model	Burner Type	Number Used in Study
1	Perkin-Elmer	303	Premix*	16
2	Perkin-Elmer	303	Boling	23
3	Beckman, DU	1300	Premix	2
4	Jarrell Ash	82-500	Premix	1
5	Jarrell Ash	82-500	Hetco	5
6	Unicam	SP-90	Premix	1
7	Evans	EEL	Premix	1
8	Hilger & Watts	——	Premix	1
9	Techtron	AA-4	——	1
10	Perkin-Elmer	290	Premix	1
11	Beckman, DB	1301	Premix	3
12	Beckman, DBG	1301	Premix	3
13	Jarrell Ash	82-600	Premix	1

* Also known as a laminar flow burner.

RESULTS

ZINC (Table 3, Figures 1, 2, and 3)

As in the analysis of nearly all other metals presented in this report, thirteen different combinations of burners and instruments were used to measure zinc, but only two were used by enough participants to permit statistical comparison of the results. The results obtained by using three other types of instruments are also presented, but the statistical significance of the latter data is questionable since the results from only five laboratories in one case, and from three in each of the other two cases were used to calculate the presented statistical measures.

Fourteen laboratories used a Perkin-Elmer Model 303 atomic absorption spectrophotometer equipped with a premix burner and 23 used identical instruments equipped with Boling burners. In both cases, the aqueous samples were aspirated directly into the burner and were not concentrated by chelation and extraction or by other means. In the analysis of sample 1 (0.050 mg/l), significantly more accurate results were obtained by use of the Boling burner (relative error 9.33 vs. 30.83), but the precision in both cases was poor and not significantly different (relative standard deviations 45.77 vs. 30.68). In the case of sample 2 (0.500 mg/l), the precision of the Boling burner was significantly better (3.06 vs. 6.73 percent), but either burner gave very good accuracy (1.36 vs. 2.30 percent).

Table 3. Summary of Data on Aqueous Zinc

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.050 mg/liter								
P.E., 303, Premix	14	2	0.0654	+0.015	0.0201	30.83	30.68	0.0635
P.E., 303, Boling	23	2	0.0453	-0.005	0.0207	9.33	45.77	0.0565
J.A., (82-500), Hetco	5	0	0.0522	+0.002	0.0138	4.40	26.42	0.0700
B., DB, Premix	3	0	0.0587	+0.009	0.0597	17.33	98.66	0.5740
B., DBG, Premix	3	0	0.0637	+0.014	0.0142	27.33	22.23	0.1404
Overall	57	6	0.0547	+0.005	0.0229	9.41	41.86	0.0644
Sample 2, 0.500 mg/liter								
P.E., 303, Premix	14	0	0.5115	+0.012	0.0344	2.30	6.73	0.1037
P.E., 303, Boling	19	4	0.5068	+0.007	0.0155	1.36	3.06	0.0432
J.A., (82-500), Hetco	5	0	0.4758	-0.024	0.0534	4.84	11.23	0.2713
B., DB, Premix	3	0	0.4733	-0.027	0.2040	5.33	43.11	2.0233
B., DBG, Premix	3	0	0.5033	+0.003	0.0562	0.67	11.17	0.5573
Overall. . . .	56	8	0.5021	+0.002	0.0409	0.41	8.15	0.0978
Sample 3, 1.000 mg/liter								
P.E., 303, Premix	14	0	1.0099	+0.010	0.0674	0.99	6.68	0.2031
P.E., 303, Boling	23	4	1.0010	-0.001	0.0512	0.10	5.12	0.1425
J.A., (82-500), Hetco	5	0	0.9684	-0.032	0.0915	3.16	9.45	0.4649
B., DB, Premix	3	0	1.4100	+0.410	0.3214	41.00	22.79	3.1870
B., DBG, Premix	3	0	1.0733	+0.073	0.1704	7.33	15.88	1.6896
Overall	56	7	1.0303	+0.030	0.1084	3.03	10.52	0.2585

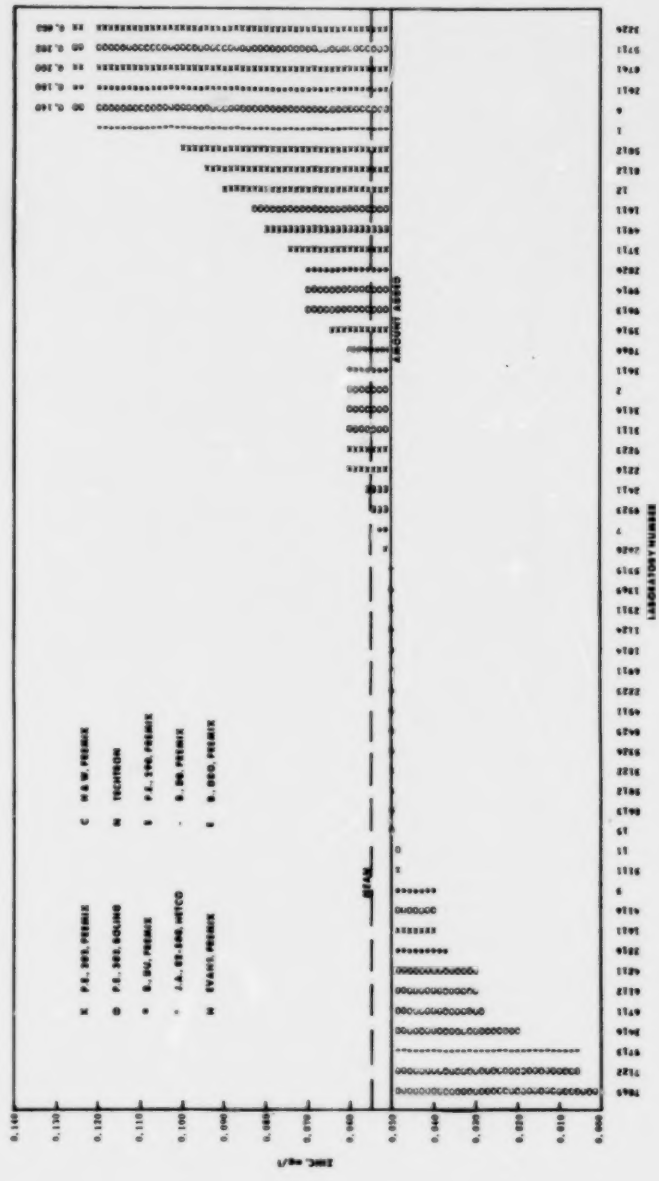

Figure 1. Bar graph for aqueous zinc in sample 1.

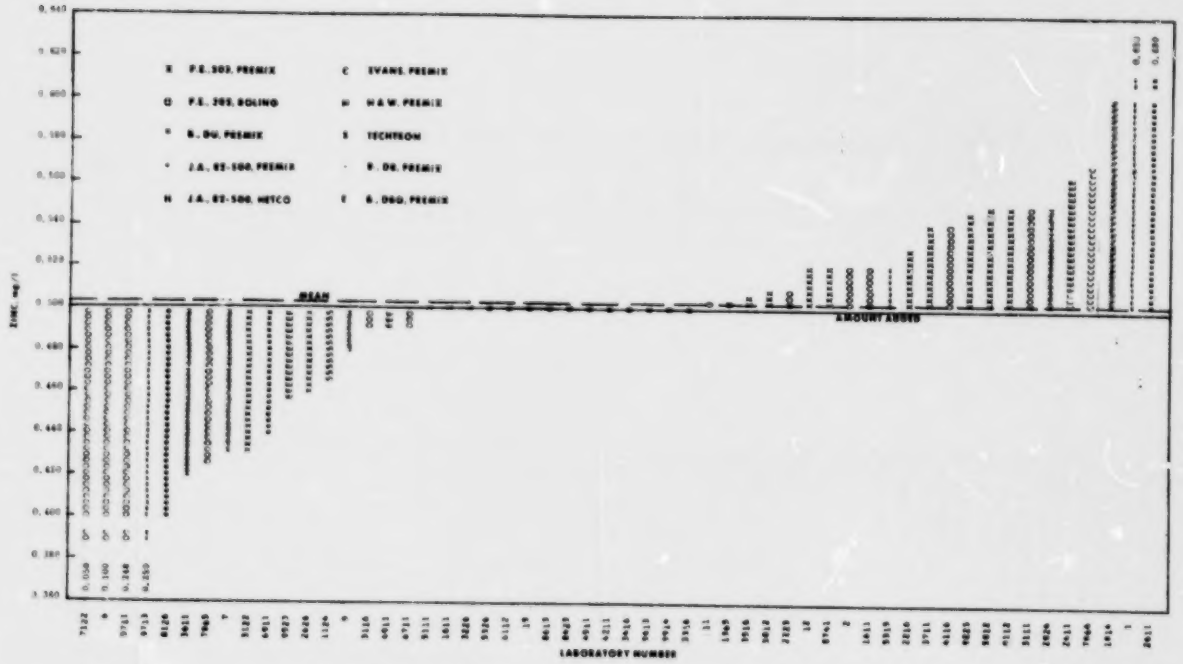

Figure 2. Bar graph for aqueous zinc in sample 2.

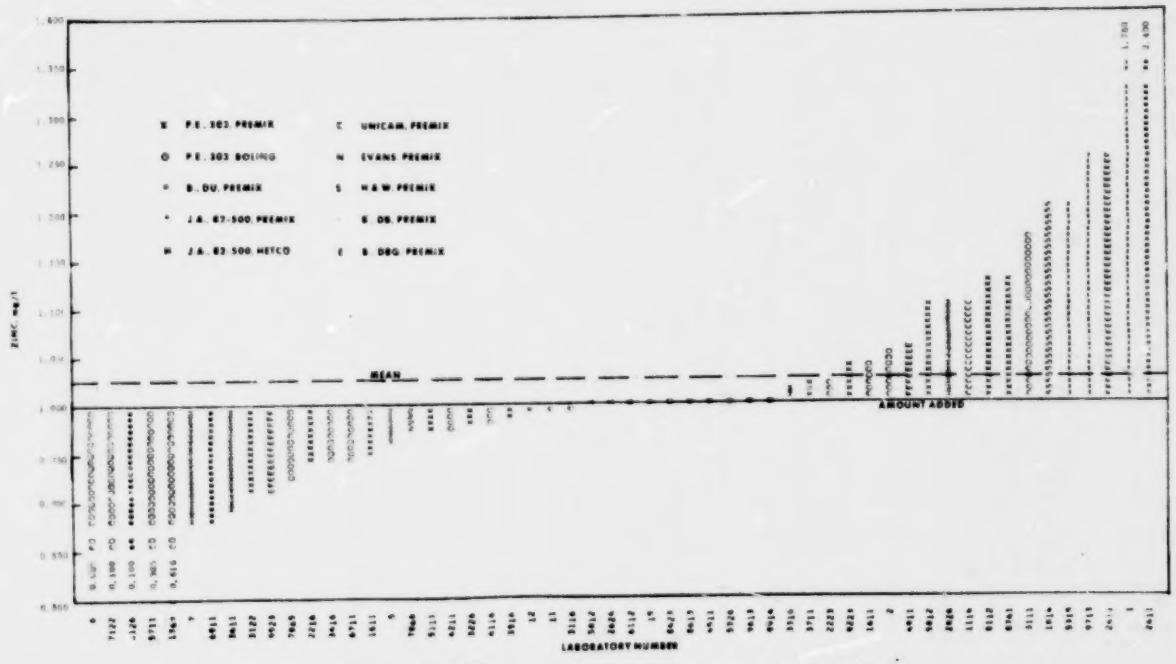

Figure 3. Bar graph for aqueous zinc in sample 3.

In the analysis of sample 3, there was no significant difference in either the precision (5.12 vs. 6.68 percent) or accuracy (0.10 and 0.99), and both were very good.

Since the drinking water standard for zinc is set at 5.0 mg/liter and, in this study, as little as 0.500 mg/liter has been determined (regardless of instrument used) by atomic absorption spectrophotometry without difficulty and with a high degree of accuracy and precision (overall 0.41 and 8.15, respectively, at the 0.5 mg/l level), it can safely be stated that this is a good and acceptable method for the determination of zinc.

CHROMIUM (Tables 4 and 5, Figures 4, 5, 6, 7, 8 and 9)

In the determination of chromium by aspiration of the aqueous sample directly into the burner (Table 4, Figures 4, 5 and 6), nine different combinations of instruments and burners were used, but only two of these were used in sufficient number to be of statistical significance.

Six laboratories used a Perkin-Elmer atomic absorption spectrophotometer equipped with a premix burner, and fourteen used an identical instrument equipped with a Boling burner. In the analysis of all three samples, there was no significant difference in the accuracy of the results obtained by the use of the two burners. In the analysis of sample 1 (0.200 mg/liter) and sample 3 (0.050 mg/liter), however, the precision of the Boling burner results were significantly better (12.26 vs. 42.56 and 26.44 vs. 51.65, respectively).

While the accuracy of all the determinations on chromium (regardless of burner used) were acceptable, only the results obtained by using the Boling burner had acceptable precision; namely, 12.26 percent at the 0.200 mg/liter level and 22.08 percent at the 0.100 mg/liter level. Since the precision at the 0.05 mg/liter level, which is the drinking water standard, was only 26.44 percent, the method is borderline in acceptability for the determination of chromium at this level.

In the determination of chromium by chelation and extraction (Table 5 and Figures 7, 8 and 9), none of the results had acceptable precision and, in general, only fair accuracy was obtained.

COPPER (Table 6, Figures 10, 11 and 12)

Thirteen different combinations of burners and instruments were used to measure copper, but only two were used by enough participants to permit statistical comparison of the results. The results obtained by using three other types of instruments are also presented, but the statistical significance of the latter data is questionable because of the few results available.

Table 4. Summary of Data on Aqueous Chromium

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.200 mg/liter					
P.E., 303, Premix	6	0	0.1962	-0.004	0.0835	1.92	42.56	0.3685
P.E., 303, Boling	14	0	0.1941	-0.006	0.0238	2.96	12.26	0.0716
Overall	30	0	0.1948	-0.05	0.0507	2.60	26.05	0.1293
			Sample 2, 0.100 mg/liter					
P.E., 303, Premix	6	0	0.1055	+0.006	0.0327	5.50	30.97	0.1442
P.E., 303, Boling	14	0	0.0971	-0.003	0.0214	2.93	22.08	0.0646
Overall	30	0	0.1048	+0.005	0.0270	4.80	25.72	0.0687
			Sample 3, 0.050 mg/liter					
P.E., 303, Premix	6	0	0.0548	+0.005	0.0283	9.67	51.65	0.1250
P.E., 303, Boling	14	0	0.0489	-0.001	0.0129	2.29	26.44	0.0389
Overall	30	0	0.0530	+0.003	0.0165	6.00	31.21	0.0422

Table 5. Summary of Data on Extracted Chromium

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.200 mg/liter					
P.E., 303, Premix	8	0	0.1660	-0.034	0.1397	17.00	84.13	0.5212
P.E., 303, Boling	7	0	0.1506	-0.049	0.0826	24.71	54.85	0.3309
Overall	19	0	0.1881	-0.012	0.1296	5.97	68.89	0.3607
			Sample 2, 0.100 mg/liter					
P.E., 303, Premix	8	0	0.0814	-0.019	0.0522	18.63	64.10	0.1947
P.E., 303, Boling	7	0	0.0803	-0.020	0.0610	19.71	76.04	0.2446
Overall	19	1	0.0819	-0.018	0.0549	18.06	66.97	0.1547
			Sample 3, 0.050 mg/liter					
P.E., 303, Premix	8	0	0.0393	-0.011	0.0299	21.50	76.10	0.1115
P.E., 303, Boling	7	0	0.0607	+0.011	0.0518	21.43	85.30	0.2075
Overall	19	1	0.0451	-0.005	0.0395	9.89	87.73	0.1114

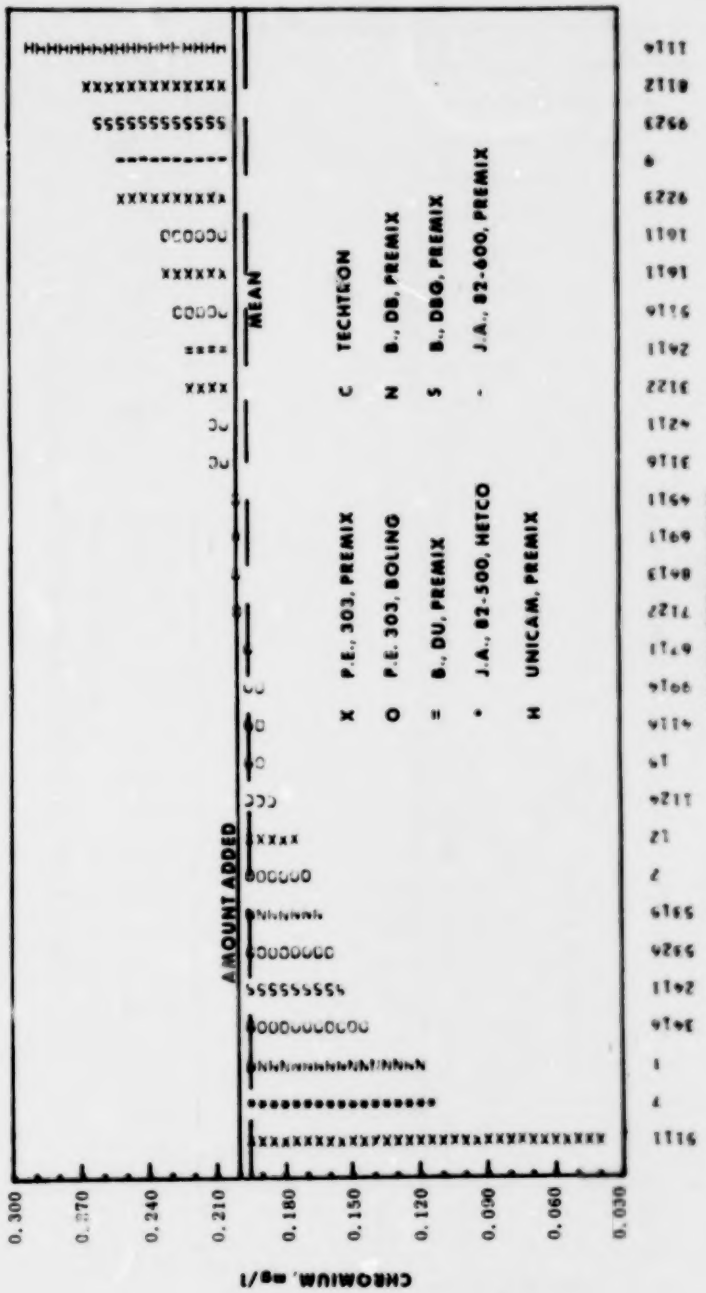

Figure 4. Bar graph for aqueous chromium in sample 1.

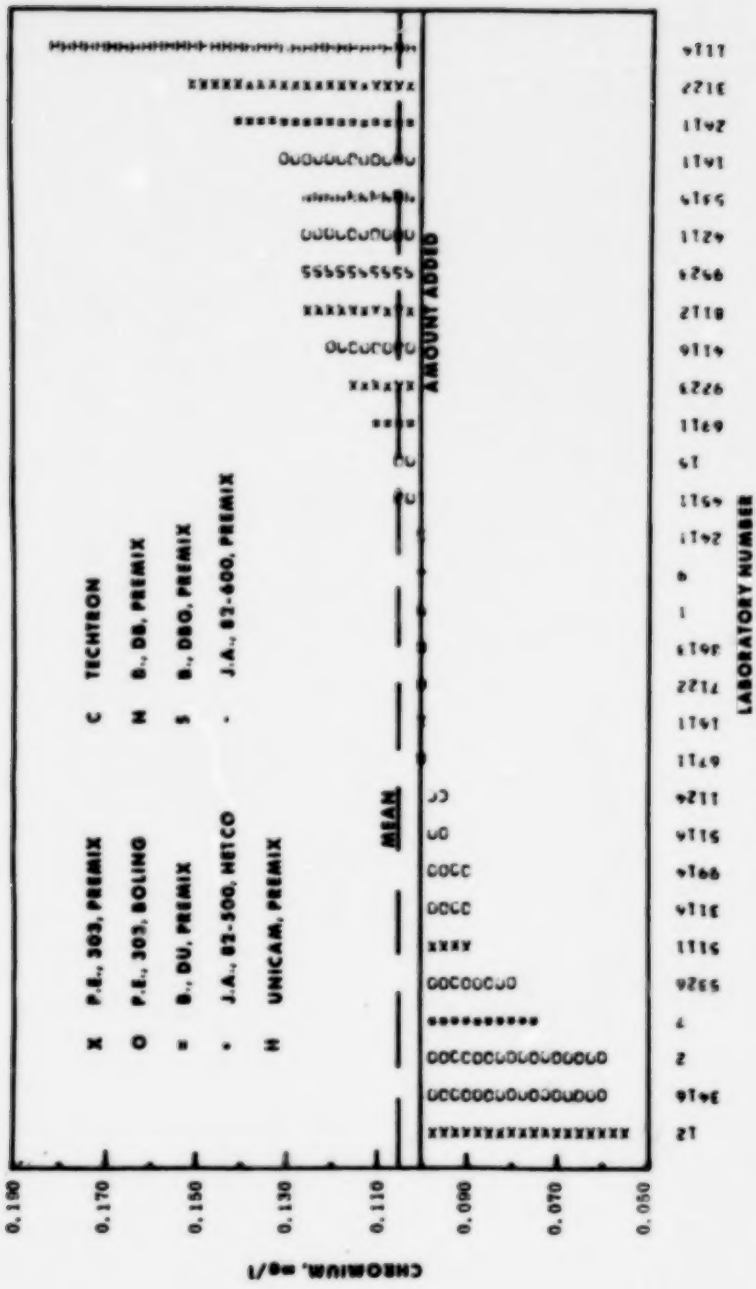

Figure 5. Bar graph for aqueous chromium in sample 2.

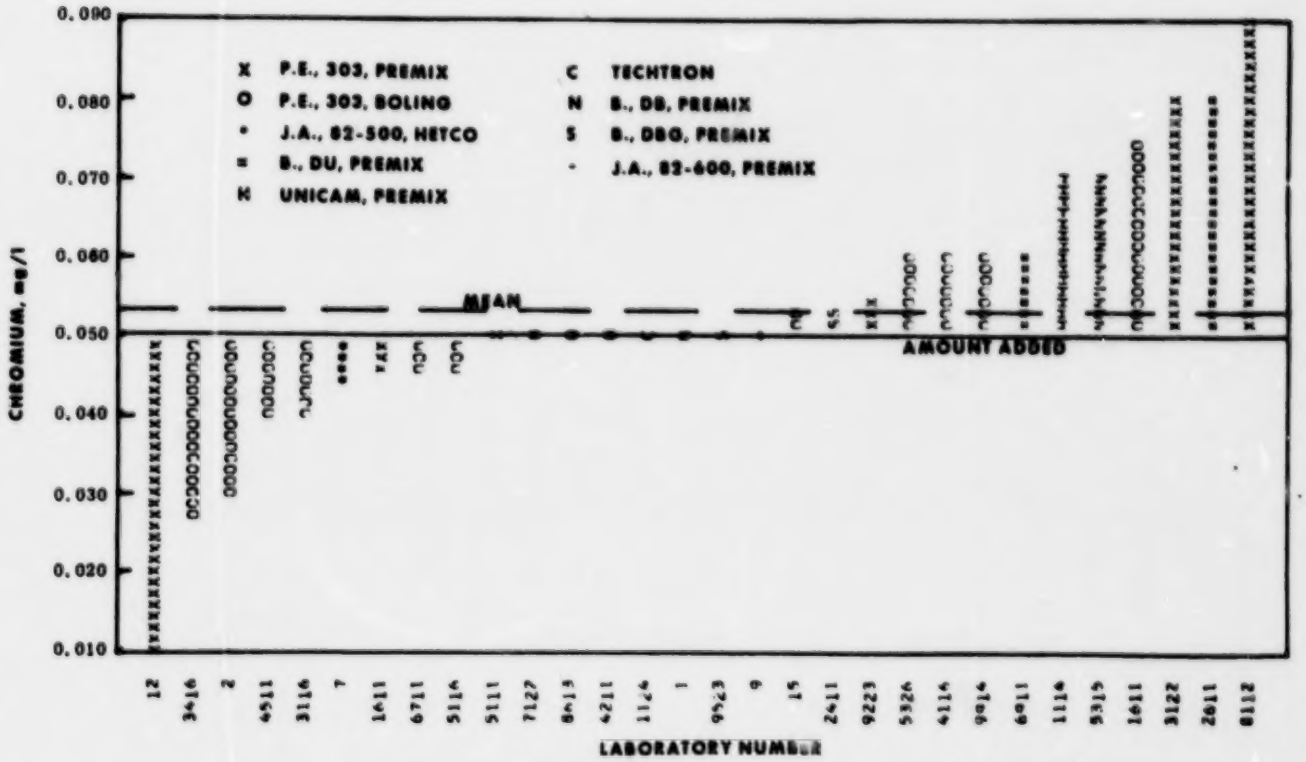

Figure 6. Bar graph for aqueous chromium in sample 3.

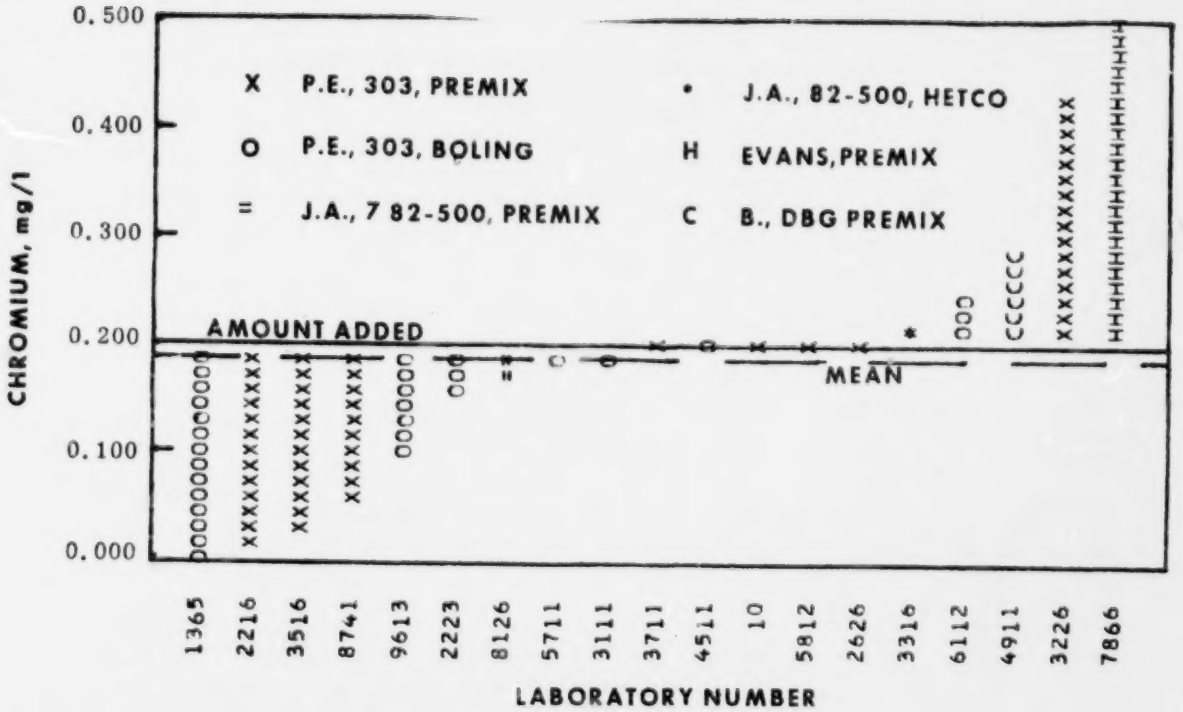

Figure 7. Bar graph for extracted chromium in sample 1.

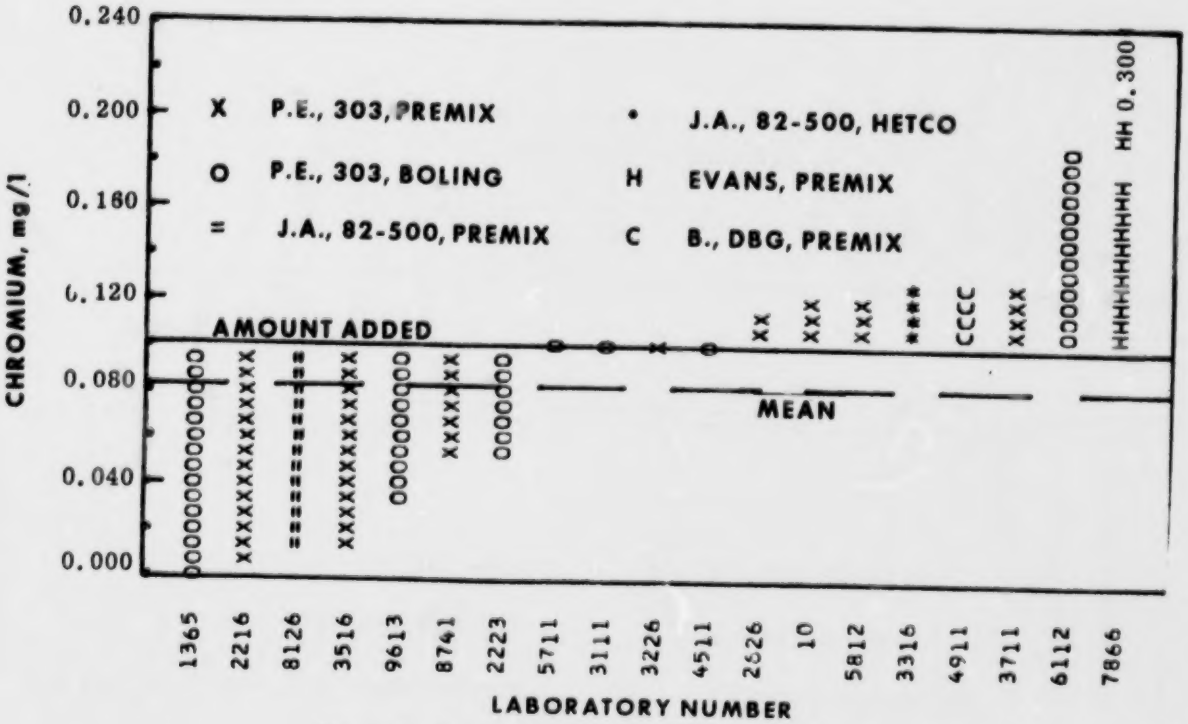

Figure 8. Bar graph for extracted chromium in sample 2.

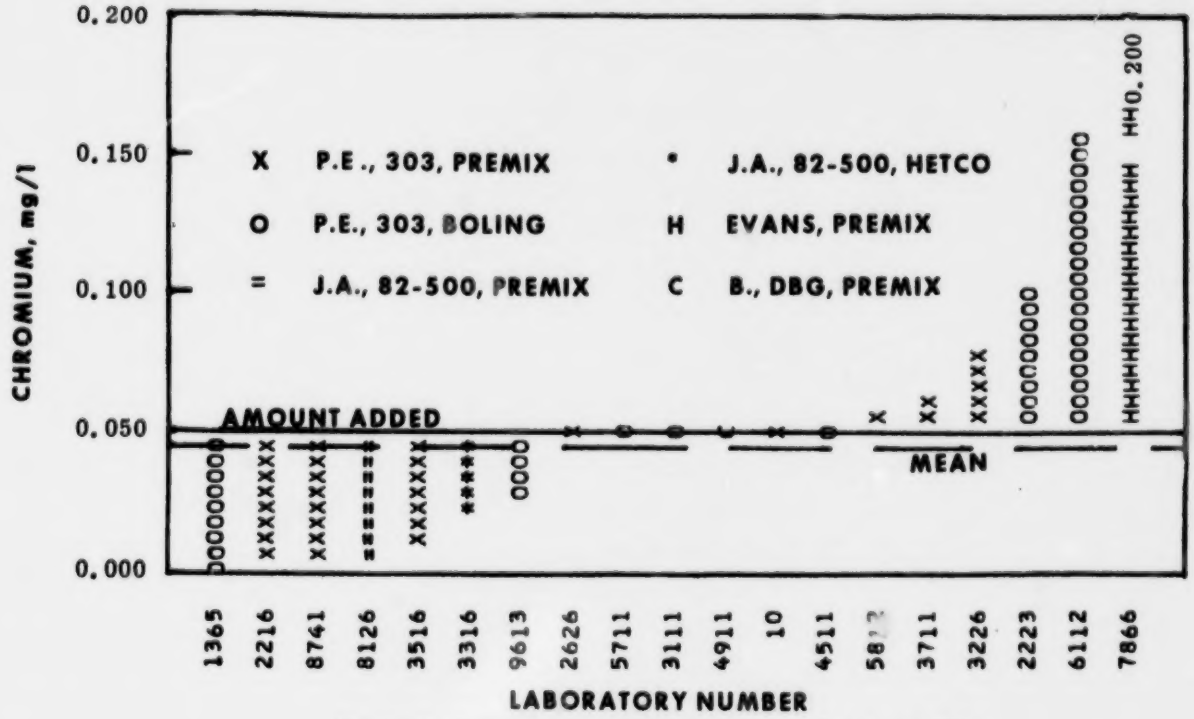

Figure 9. Bar graph for extracted chromium in sample 3.

Table 6. Summary of Data on Aqueous Copper

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.050 mg/liter								
P.E., 303, Premix	16	0	0.0809	+0.031	0.0326	61.75	40.26	0.0945
P.E., 303, Boling	23	1	0.0541	+0.004	0.0227	8.27	42.00	0.0613
J.A., (82-500), Hetco	5	0	0.0466	-0.003	0.0317	6.80	68.04	0.1610
B., DB, Premix	3	0	0.0607	+0.011	0.0090	21.33	14.87	0.0894
B., DBG, Premix	3	0	0.0620	+0.012	0.0120	24.00	19.35	0.1190
Overall	58	1	0.0668	+0.017	0.0316	33.61	47.35	0.0742
Sample 2, 0.250 mg/liter								
P.E., 303, Premix	16	0	0.2929	+0.043	0.0532	17.15	18.18	0.1546
P.E., 303, Boling	23	3	0.2555	+0.006	0.0195	2.20	7.63	0.0537
J.A., (82-500), Hetco	5	0	0.2944	+0.044	0.1005	17.76	34.14	0.5104
B., DB, Premix	3	1	0.2795	+0.030	0.0007	11.80	0.25	0.0266
B., DBG, Premix	3	0	0.2670	+0.017	0.0507	6.80	18.98	0.5026
Overall	59	2	0.2712	+0.021	0.0652	8.48	24.06	0.1530
Sample 3, 1.000 mg/liter								
P.E., 303, Premix	16	0	1.0539	+0.054	0.1139	5.39	10.80	0.3305
P.E., 303, Boling	23	5	1.0062	+0.006	0.0597	0.62	5.93	0.1683
J.A., (82-500), Hetco	5	0	1.0310	+0.031	0.0532	3.10	5.16	0.2703
B., DB, Premix	3	1	1.2400	+0.240	0.0141	24.00	1.14	0.5328
B., DBG, Premix	3	0	1.0383	+0.038	0.1790	3.83	17.24	1.7754
Overall	59	6	1.0342	+0.034	0.1162	3.42	11.23	0.2746

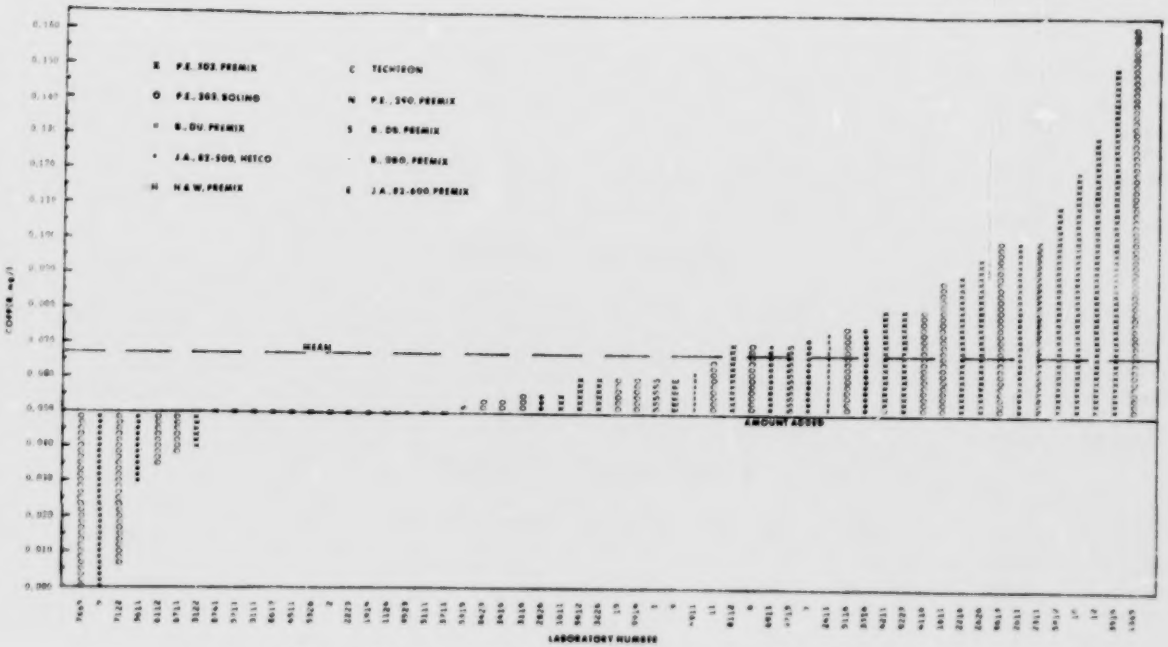

Figure 10. Bar graph for aqueous copper in sample 1.

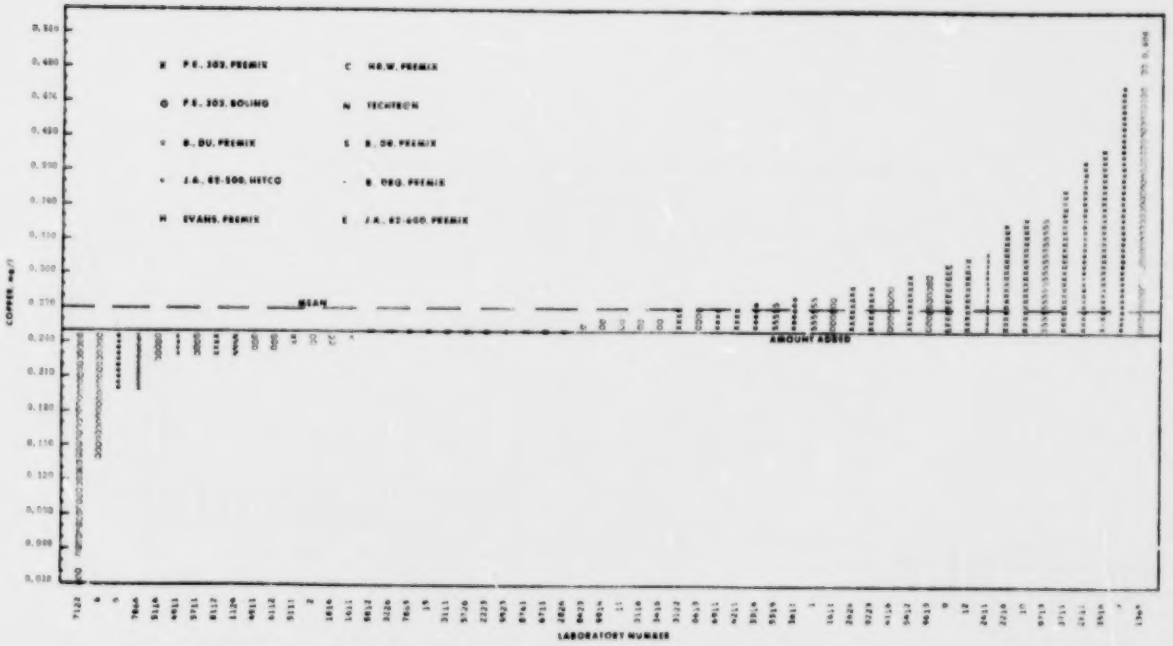

Figure 11. Bar graph for aqueous copper in sample 2.

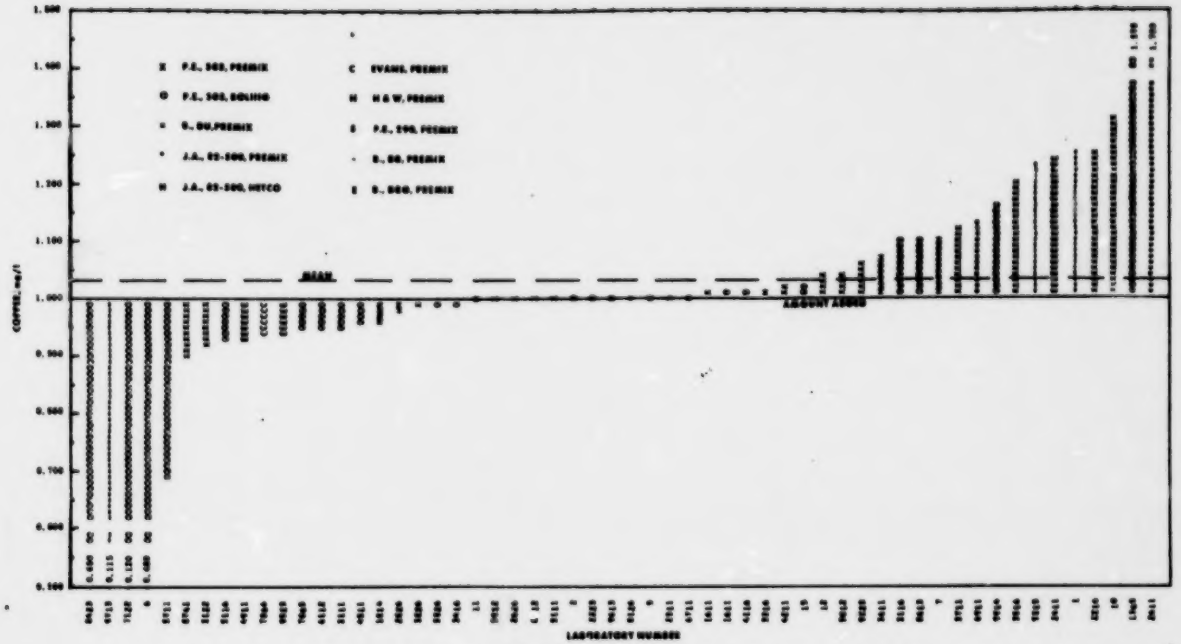

Figure 12. Bar graph for aqueous copper in sample 3.

Sixteen laboratories used a Perkin-Elmer atomic absorption spectrophotometer equipped with a premix burner and 23 used an identical instrument equipped with a Boling burner. In both cases, the aqueous samples were aspirated directly into the burner and were not concentrated by chelation and extraction or other means. In the analysis of sample 1 (0.050 mg/liter), significantly more accurate results were obtained by the use of the Boling burner, but the precision in both cases was poor and not significantly different (relative standard deviation 42.00 vs. 40.26). In the case of sample 2 (0.250 mg/liter), both the accuracy (2.20 vs. 17.15) and the precision (7.63 vs. 18.18) were better with the Boling burner. In the analysis of sample 3 (1.000 mg/liter), as with sample 2, both the accuracy (0.62 vs. 5.39) and the precision (5.93 vs. 10.80) were better with the Boling burner.

Since the drinking water standard for copper is set at 1.00 mg/liter and, in this study, this level (regardless of instrument used) has been determined with a high degree of accuracy and precision (overall 3.42 and 11.23, respectively), it can safely be stated that this is a good and acceptable method for the determination of copper at this level.

MAGNESIUM (Table 7, Figures 13, 14 and 15)

As with most of the other metals, thirteen different combinations of burners and instruments were used in analyzing for magnesium, but only two were used by enough participants to permit statistical comparison of the results.

Fourteen laboratories used a Perkin-Elmer atomic absorption spectrophotometer equipped with a premix burner and seventeen used an identical instrument equipped with a Boling burner. In both cases, the aqueous samples were aspirated directly into the burner and were not concentrated by chelation and extraction or other means. In the analysis of sample 1 (0.200 mg/liter), there was no significant difference in either the accuracy (4.96 vs. 5.57) or the precision (6.55 vs. 10.35) obtained by using the two types of burners. In the case of sample 2 (0.100 mg/liter), there was no significant difference (8.15 vs. 20.14) in the accuracy obtained by using the two different burners, but the precision obtained using the Boling burner was significantly better (10.29 vs. 22.89 percent). In the analysis of sample 3 (0.050 mg/liter), there was no significant difference in either the accuracy (8.50 vs. 8.20) or the precision (10.16 vs. 9.91) obtained by using the two different types of burners.

The overall accuracy and precision (regardless of the instrument used) at the 0.05 and 0.1 mg/liter level were not as good as desired, but at the 0.200 mg/liter level were very acceptable. Since there is no drinking water standard established for magnesium, this method certainly is sensitive enough and is acceptable for the determination of magnesium.

Table 7. Summary of Data on Aqueous Magnesium

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.200 mg/liter								
P.E., 303, Premix	14	0	0.2111	+0.011	0.0219	5.57	10.35	0.0659
P.E., 303, Boling	17	3	0.2099	+0.010	0.0137	4.96	6.55	0.0414
J.A., (82-500), Hetco	3	0	0.2253	+0.025	0.0042	12.67	1.85	0.0413
B., DBG, Premix	3	0	0.2507	+0.051	0.0490	25.33	19.55	0.4859
Overall	47	5	0.2126	+0.013	0.0223	6.30	10.49	0.0542
Sample 2, 0.100 mg/liter								
P.E., 303, Premix	14	0	0.1201	±0.020	0.0275	20.14	22.89	0.0828
P.E., 303, Boling	17	4	0.1082	+0.008	0.0111	8.15	10.29	0.0343
J.A., (82-500), Hetco	3	0	0.1807	+0.081	0.0956	80.67	52.90	0.9477
B., DBG, Premix	3	0	0.1183	+0.018	0.0126	18.33	10.63	0.1248
Overall	47	4	0.1129	+0.013	0.0306	12.93	27.10	0.0741
Sample 3, 0.050 mg/liter								
P.E., 303, Premix	13	3	0.0541	+0.004	0.0054	8.20	9.91	0.0181
P.E., 303, Boling	17	5	0.0543	+0.004	0.0055	8.50	10.16	0.0174
J.A., (82-500), Hetco	3	0	0.0610	+0.011	0.0254	22.00	41.57	0.2514
B., DBG, Premix	3	0	0.0597	+0.010	0.0055	19.33	9.23	0.0546
Overall	45	2	0.0598	+0.010	0.0218	19.53	36.42	0.0527

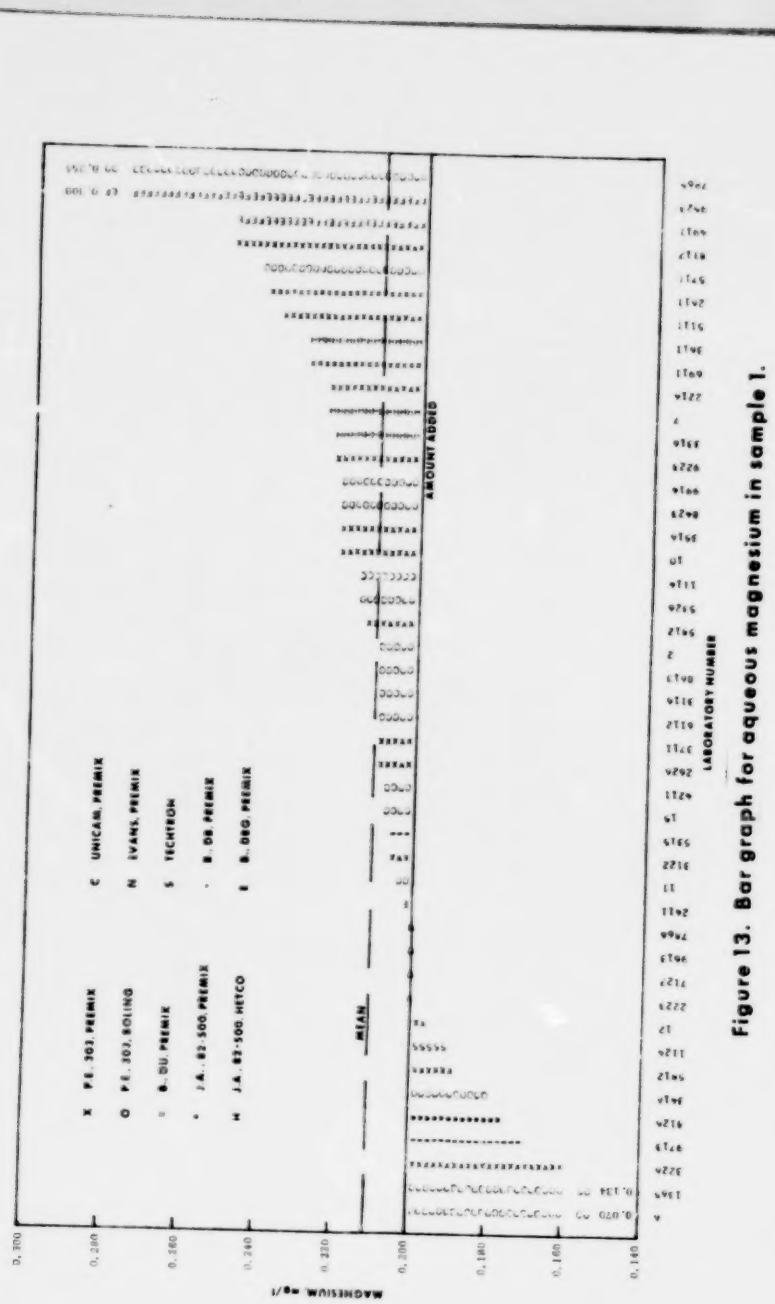

Figure 13. Bar graph for aqueous magnesium in sample 1.

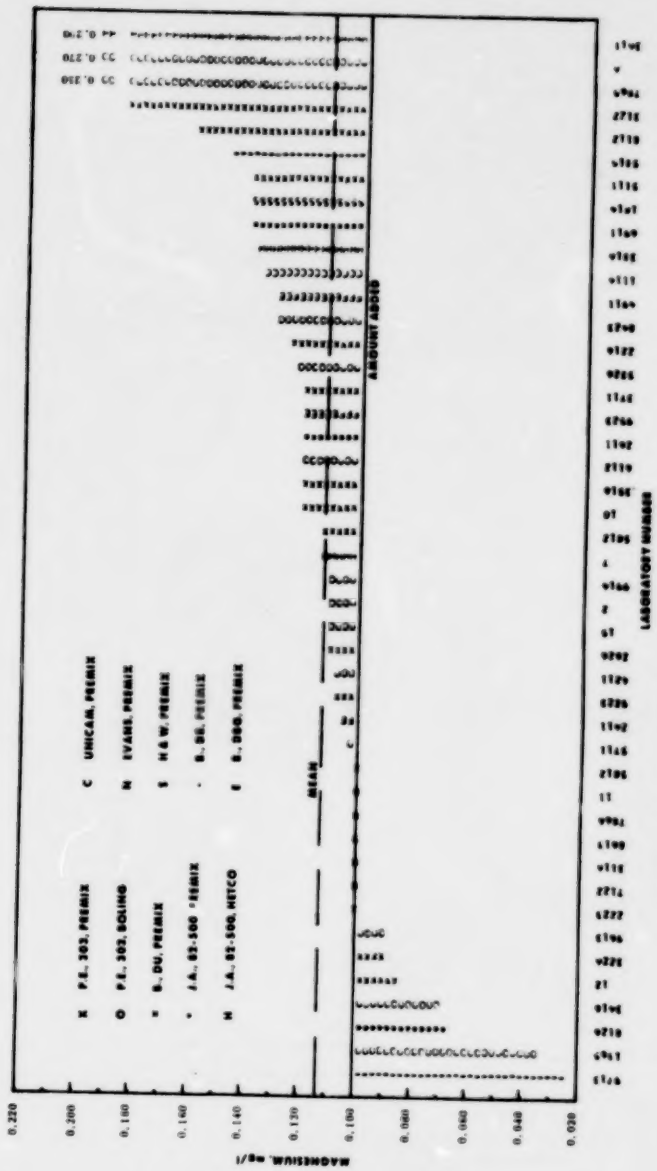

Figure 14. Bar graph for aqueous magnesium in sample 2.

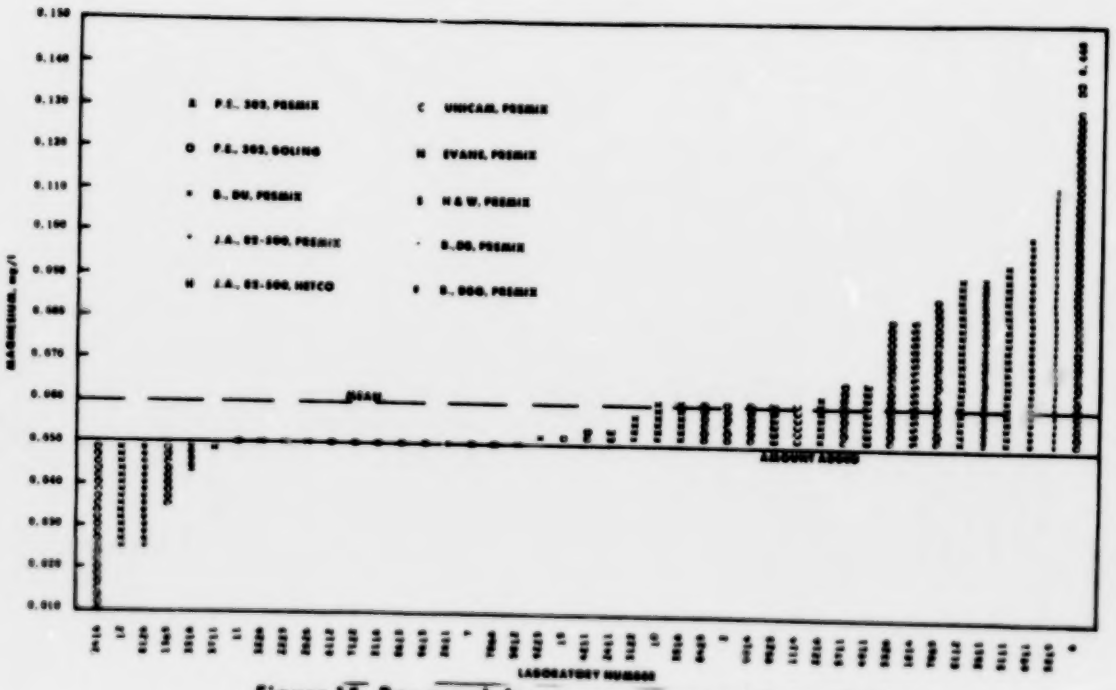

Figure 15. Bar graph for aqueous magnesium in sample 3.

MANGANESE (Tables 8 and 9, Figures 16, 17, 18, 19, 20 and 21)

In the determination of manganese by direct aspiration of the aqueous sample into the burner (Table 8, Figures 16, 17 and 18), thirteen different combinations of burners and instruments were used, but only two were used by enough participants to permit statistical comparison of the results. The results obtained by using three other types of instruments are presented, but the statistical significance of the data is questionable because of the few results available.

Nine laboratories used a Perkin-Elmer spectrophotometer equipped with premix burner and fourteen, an identical instrument equipped with a Boling burner. In the analysis of sample 1 (0.050 mg/liter), there was no significant difference in the accuracy (6.00 vs. 9.75) of the data obtained by using the two types of burners, but the precision obtained with the Boling burner was significantly better (13.50 vs. 31.40 percent). In the analysis of sample 2 (0.500 mg/liter), again the accuracies were comparable, but the precision was significantly better with the Boling burner (8.43 vs. 14.93).

In the analysis of sample 3 (0.250 mg/liter), the accuracy and precision were equally good. Since the drinking water standard is set at 0.05 mg/liter, the results of this study indicate that manganese can be determined at this level with an acceptable degree of accuracy and precision only if a Boling burner is used.

In the determination of manganese by chelation and extraction and then aspiration into the flame (Table 9 and Figures 19, 20 and 21), too few laboratories submitted data to be of statistical significance, but that which was submitted would indicate that this procedure is not satisfactory for the determination of manganese.

SILVER (Tables 10 and 11, Figures 22, 23, 24, 25, 26 and 27)

In the determination of silver by direct aspiration of the aqueous sample into the burner (Table 10, Figures 22, 23 and 24), eight participants used a Perkin-Elmer spectrophotometer equipped with Boling burner and achieved acceptable accuracy and precision in the analysis of all three samples. Since the drinking water standard is set at 0.050 mg/liter and the accuracy and precision at this level was found to be respectively 10.57 and 17.47 percent, this is an acceptable method of analysis for silver.

In the determination of silver by chelation and extraction before aspirating into the flame (Table 11, Figures 25, 26 and 27), too few laboratories submitted data to make the calculated results of statistical significance, but that which is available would indicate that the procedure is unacceptable.

Table 8. Summary of Data on Aqueous Manganese

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.050 mg/liter					
P.E., 303, Premix	9	1	0.0549	+0.005	0.0172	9.75	31.40	0.0643
P.E., 303, Boling	14	0	0.0530	+0.003	0.0072	6.00	13.50	0.0216
J.A., (82-500), Hetco	5	0	0.0994	+0.049	0.0702	98.80	70.58	0.3563
B., DB, Premix	3	0	0.0500	0.000	0.0000	0.00	0.00	0.0000
B., DBG, Premix	3	0	0.0547	+0.005	0.0203	9.33	37.15	0.2014
Overall	41	5	0.0539	+0.004	0.0138	7.78	25.58	0.0342
			Sample 2, 0.500 mg/liter					
P.E., 303, Premix	9	0	0.5462	+0.046	0.0815	9.24	14.93	0.2880
P.E., 303, Boling	14	0	0.5066	+0.007	0.0427	1.33	8.43	0.1287
J.A., (82-500), Hetco	5	0	0.4534	-0.047	0.0909	9.32	20.05	0.4618
B., DB, Premix	3	0	0.5307	+0.031	0.0977	6.13	18.41	0.9686
B., DBG, Premix	3	0	0.4600	-0.040	0.0312	8.00	6.79	0.3096
Overall	42	5	0.5111	+0.011	0.0539	2.22	10.56	0.1334
			Sample 3, 0.250 mg/liter					
P.E., 303, Premix	9	1	0.2459	-0.004	0.0157	1.60	6.38	0.0586
P.E., 303, Boling	14	0	0.2610	+0.011	0.0304	4.40	11.65	0.0916
J.A., (82-500), Hetco	5	0	0.2224	-0.028	0.0608	11.04	27.33	0.3087
B., DB, Premix	3	0	0.2640	+0.014	0.0441	5.60	16.72	0.4377
B., DBG, Premix	3	0	0.2360	-0.014	0.0213	5.60	9.02	0.2111
Overall	42	2	0.2505	0.000	0.0409	0.18	16.33	0.1000

Table 9. Summary of Data on Extracted Manganese

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.050 mg/liter					
P.E., 303, Premix	4	0	0.0515	+0.002	0.0206	3.00	40.06	0.1314
P.E., 303, Boling	3	0	0.0263	-0.024	0.0414	47.33	157.05	0.4101
Overall	8	0	0.0419	-0.008	0.0289	16.25	69.09	0.1080
			Sample 2, 0.500 mg/liter					
P.E., 303, Premix	4	0	0.3478	-0.152	0.2768	30.45	79.60	1.7633
P.E., 303, Boling	2	0	0.0750	-0.425	0.1061	85.00	141.42	3.9950
Overall	7	0	0.2516	-0.248	0.2386	49.69	94.83	0.9559
			Sample 3, 0.250 mg/liter					
P.E., 303, Premix	4	0	0.1615	-0.089	0.1191	35.40	73.77	0.7590
P.E., 303, Boling	2	0	0.0050	-0.245	0.0071	98.00	141.42	0.2664
Overall	7	0	0.1394	-0.111	0.1374	44.23	98.57	0.5507

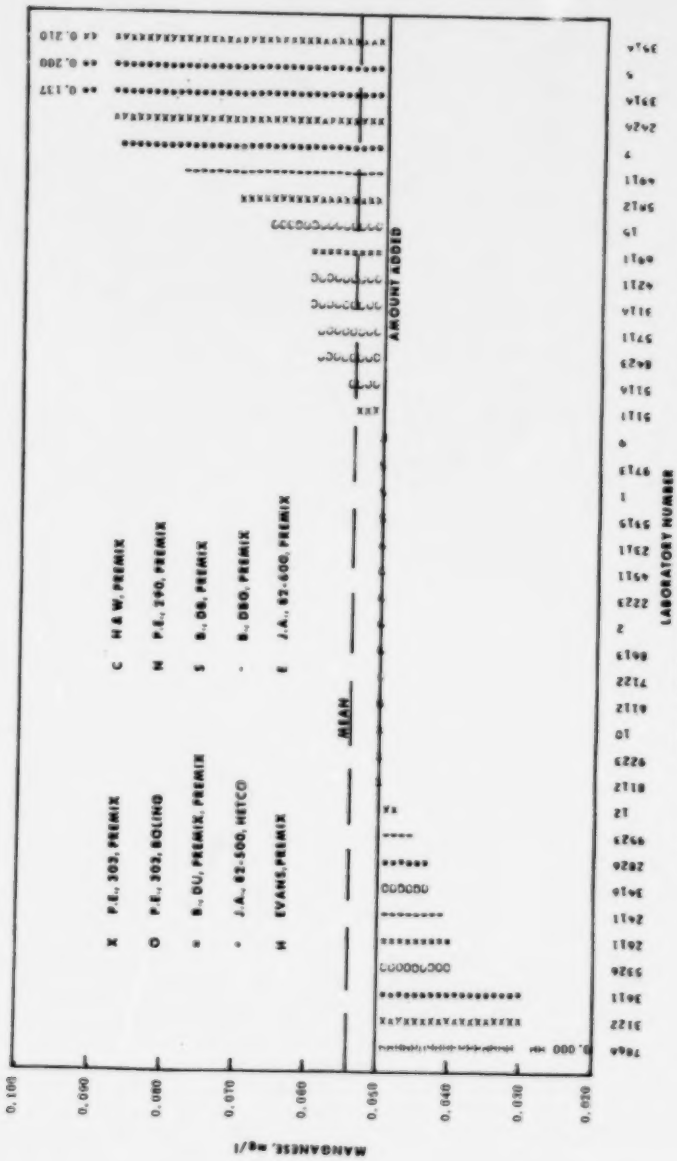

Figure 16. Bar graph for aqueous manganese in sample 1.

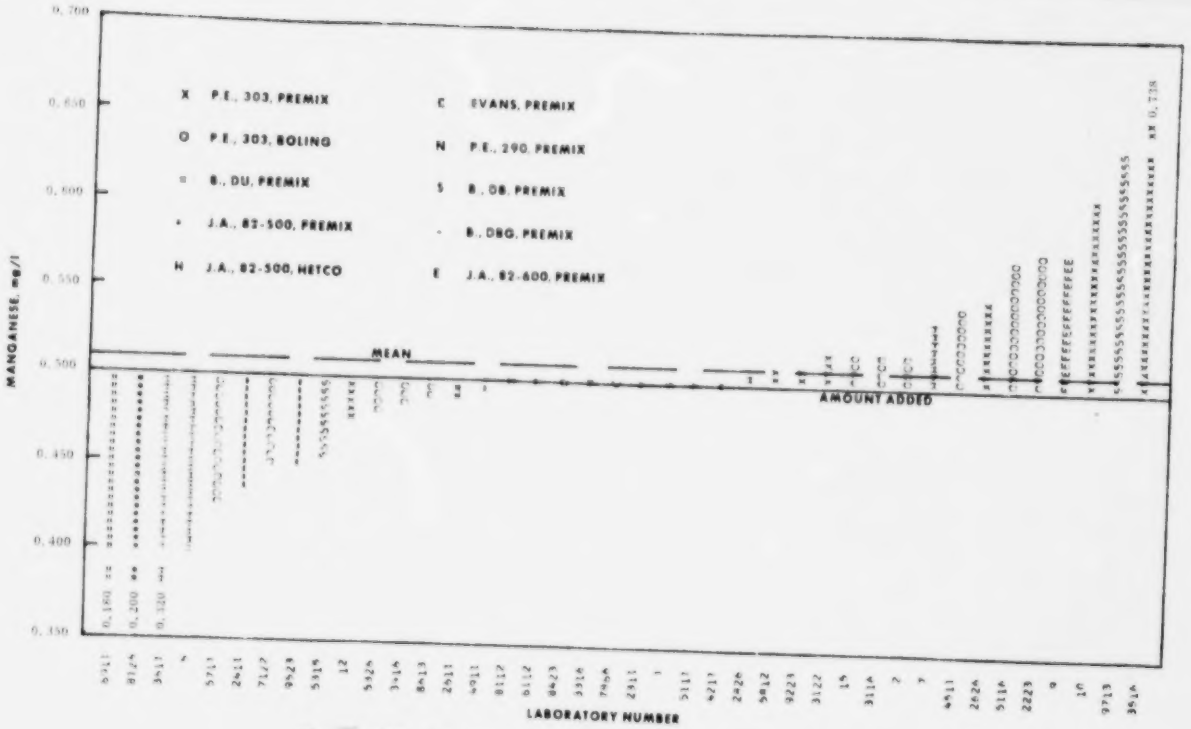

Figure 17. Bar graph for aqueous manganese in sample 2.

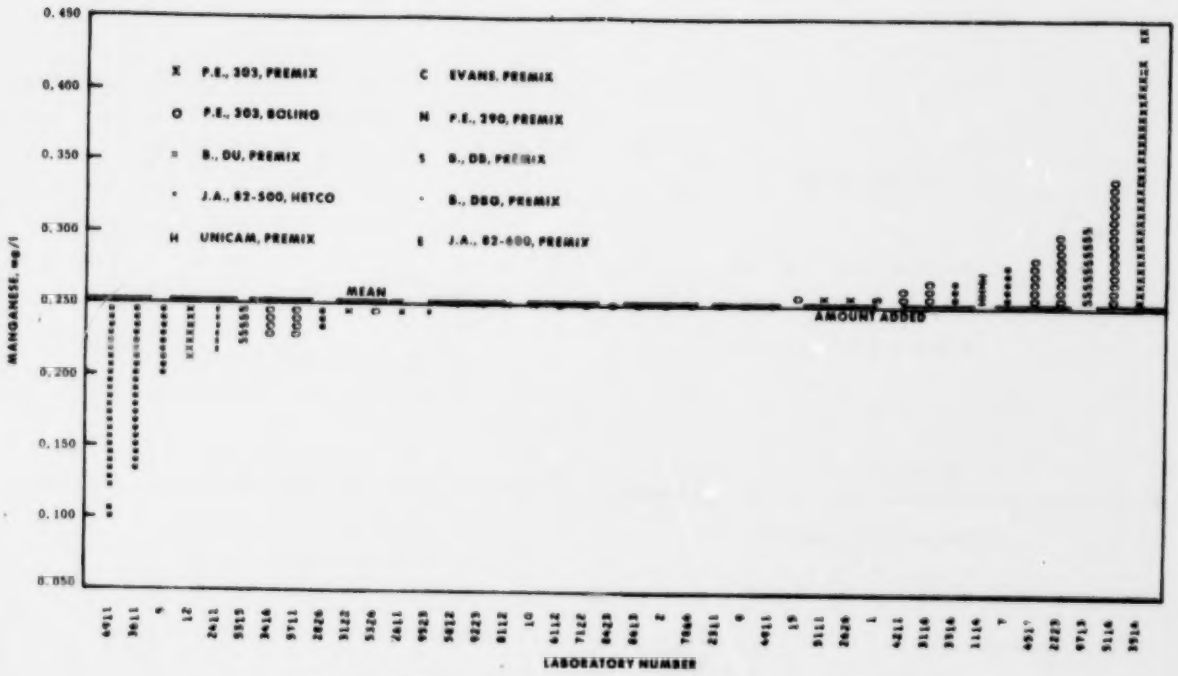

Figure 18. Bar graph for aqueous manganese in sample 3.

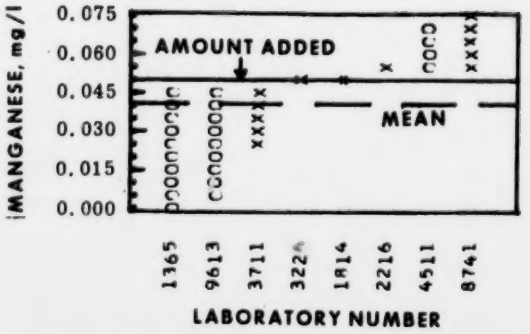

Figure 19. Bar graph for extracted manganese in sample 1.

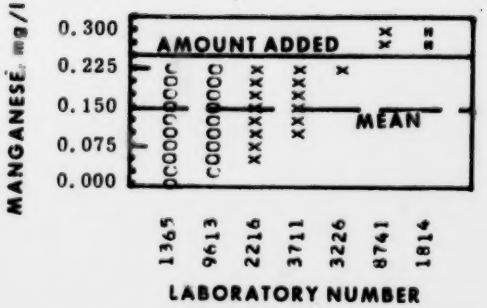

Figure 21. Bar graph for extracted manganese in sample 3.

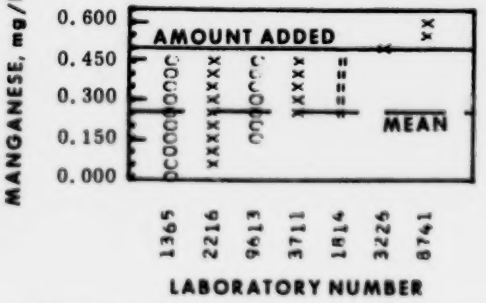

Figure 20. Bar graph for extracted manganese in sample 2.

X P.E., 303, PREMIX

O P.E., 303, BOLING

= H & W, PREMIX

Table 10. Summary of Data on Aqueous Silver

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.050 mg/liter					
P.E., 303, Boling	8	1	0.0553	+0.005	0.0097	10.57	17.47	0.0387
J.A., (82-500), Hetco	3	0	0.0720	+0.022	0.0211	44.00	29.27	0.2089
Overall	21	0	0.0656	+0.016	0.0206	31.24	31.41	0.0561
			Sample 2, 0.100 mg/liter					
P.E., 303, Boling	8	1	0.0929	-0.007	0.0099	7.14	10.70	0.0398
J.A., (82-500), Hetco	3	0	0.1237	+0.024	0.0446	23.67	36.08	0.4424
Overall	21	0	0.1067	+0.007	0.0253	6.71	23.68	0.0688
			Sample 3, 0.200 mg/liter					
P.E., 303, Boling	8	1	0.2146	+0.015	0.0162	7.29	7.55	0.0649
J.A., (82-500), Hetco	3	0	0.2667	+0.067	0.0702	33.33	26.34	0.6965
Overall	21	0	0.2336	+0.039	0.0477	19.29	20.01	0.1300

Table 11. Summary of Data on Extracted Silver

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.050 mg/liter					
P.E., 303, Premix	4	0	0.0623	+0.012	0.0277	24.50	44.49	0.1764
P.E., 303, Boling	4	0	0.1063	+0.056	0.0729	112.50	68.60	0.4643
Overall	9	0	0.0798	+0.030	0.0543	59.56	68.01	0.1913
			Sample 2, 0.100 mg/liter					
P.E., 303, Premix	4	0	0.0520	-0.048	0.0443	48.00	85.27	0.2824
P.E., 303, Boling	4	0	0.1350	+0.035	0.0934	35.00	69.16	0.5947
Overall	9	0	0.0909	-0.009	0.0761	9.11	83.72	0.2687
			Sample 3, 0.200 mg/liter					
P.E., 303, Premix	3	1	0.0087	-0.191	0.0110	95.67	126.57	0.1088
P.E., 303, Boling	4	0	0.1798	-0.020	0.1398	10.13	77.78	0.8906
Overall	9	0	0.1168	-0.083	0.1219	41.61	104.41	0.4306

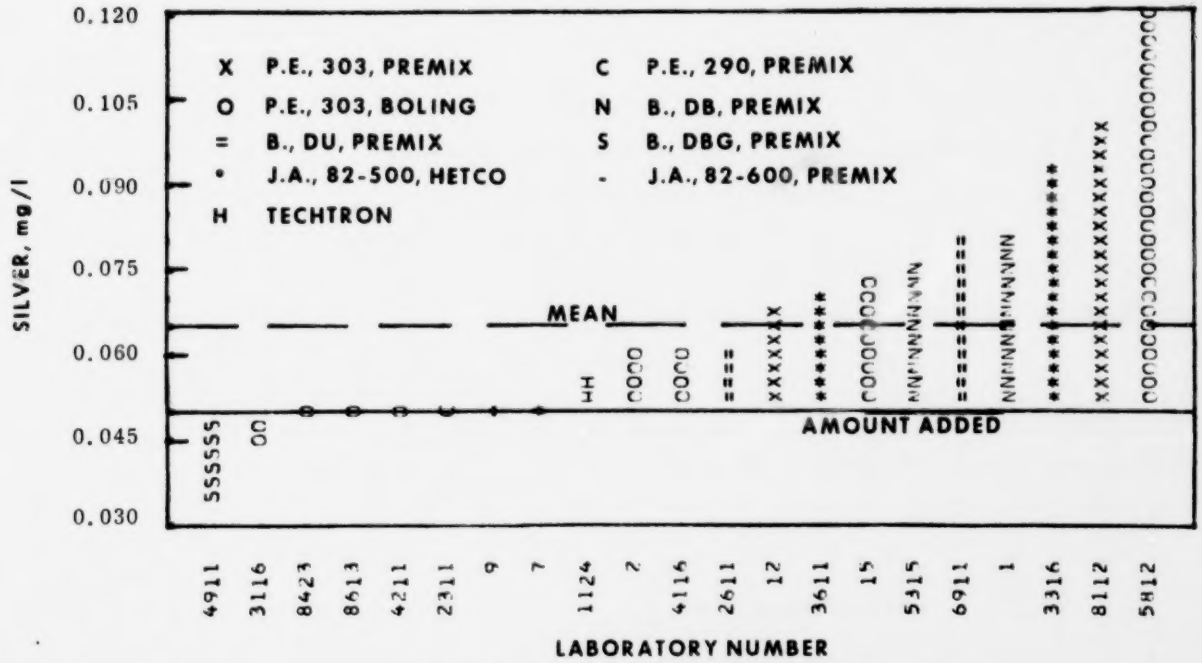

Figure 22. Bar graph for aqueous silver in sample 1.

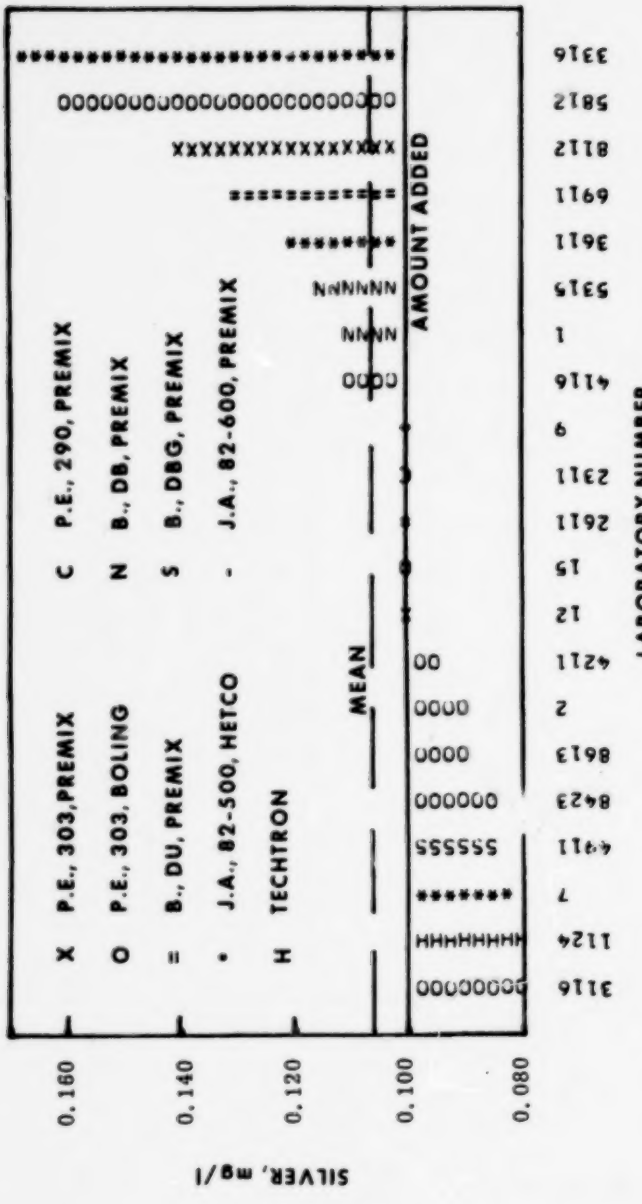

Figure 23. Bar graph for aqueous silver in sample 2.

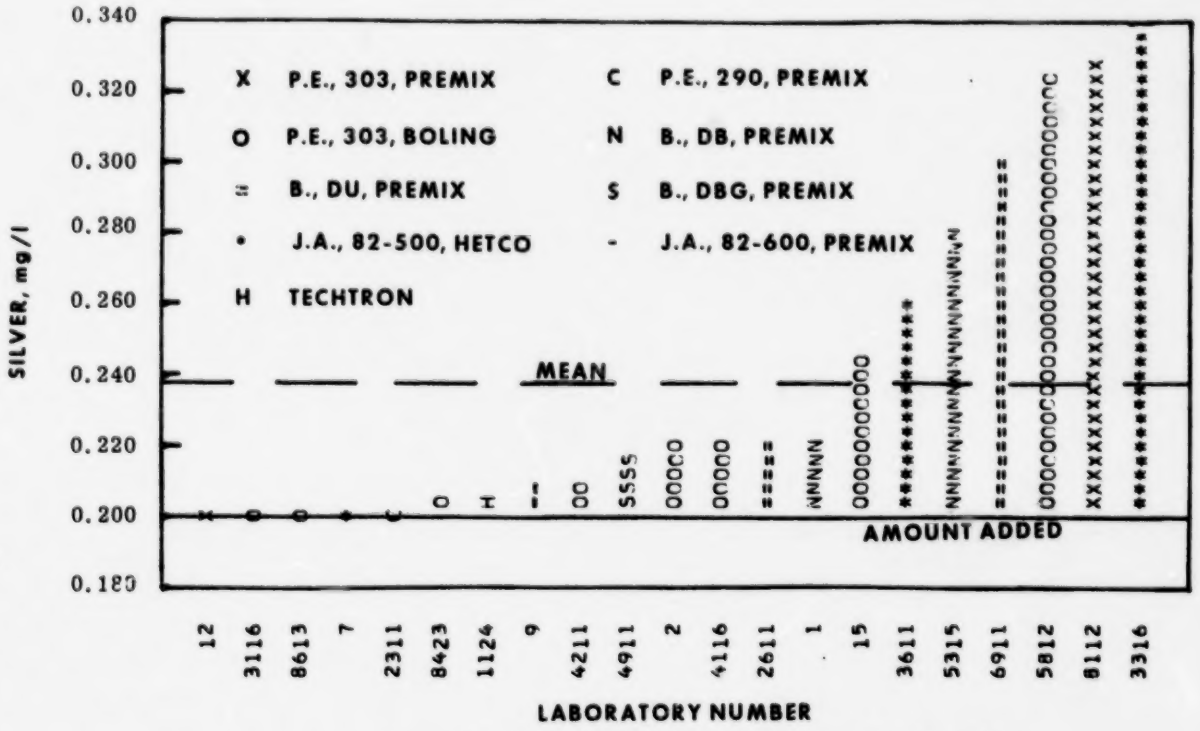

Figure 24. Bar graph for aqueous silver in sample 3.

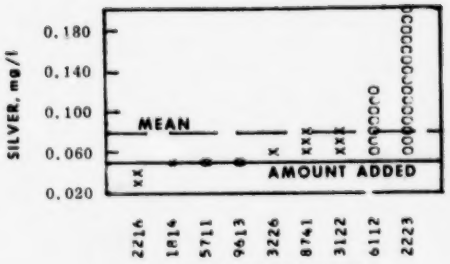

Figure 25. Bar graph for extracted silver in sample 1.

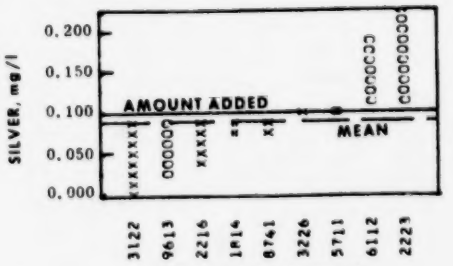

Figure 26. Bar graph for extracted silver in sample 2.

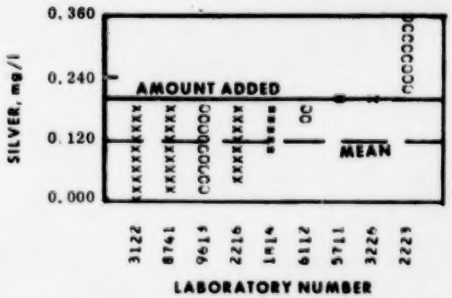

Figure 27. Bar graph for extracted silver in sample 3.

X P.E., 303, PREMIX

O P.E., 303, BOLING

= H & W, PREMIX

LEAD (Tables 12 and 13, Figures 28, 29, 30, 31, 32 and 33)

In the determination of lead by direct aspiration of the aqueous sample into the burner (Table 12, Figures 28, 29 and 30), too few laboratories analyzed the samples, using a premix burner, to make the results statistically significant, and those using a Boling burner obtained unsatisfactory precision at all levels.

In the determination of lead by chelation and extraction before aspirating into the flame (Table 13 and Figures 31, 32 and 33), nine laboratories used a Perkin-Elmer equipped with a premix burner and eight, an identical instrument with a Boling burner. In the analysis of sample 1 (0.200 mg/liter), both the accuracy and the precision were comparable, regardless of whether the premix or Boling burner was used. The same results were obtained in the analysis of sample 3 (0.050 mg/liter), but with sample 2 (0.100 mg/liter) the precision obtained using the premix burner was significantly better (21.67 vs. 55.22 percent) than that obtained with the Boling burner.

Since the drinking water standard for lead is set at 0.050 mg per liter and the accuracy and precision at this level, using the premix burner, were only 19.00 and 23.46 percent, respectively (Boling burner, 3.14 and 53.33, respectively), the method leaves something to be desired. Admittedly, because of the poor sensitivity of direct aspiration, the extraction procedure is better. Unfortunately, although the accuracy of the extraction procedure is good, the precision is rather poor.

CADMIUM (Tables 14 and 15, Figures 34, 35, 36, 37, 38 and 39)

In the determination of cadmium by direct aspiration of the aqueous sample into the burner (Table 14, Figures 34, 35 and 36), a statistically significant comparison could not be made because too few laboratories analyzed the samples with a premix burner, but, as with most of the other metals, the Boling burner appeared to give better results. For example, in the analysis of sample 2 (0.050 mg/liter), both the precision (7.77 vs. 23.47) and accuracy (2.00 vs. 21.60) were excellent, and better than were obtained with the premix burner. A similar situation occurred in the analysis of sample 3 (0.100 mg/l). Unfortunately, in the analysis of sample 1 (0.010 mg/liter), the precision and accuracy obtained by both burners were unacceptable. Since the drinking water standard is set at this same level, this method is not acceptable for the determination of cadmium at this level.

In the determination of cadmium by chelation and extraction before aspirating into the flame (Table 15, Figures 37, 38 and 39), again too few laboratories analyzed the samples to make possible any statistically significant comparisons. In general, the accuracy obtained by using the Boling burner was better than that obtained by using the premix burner and is acceptable, but poor precision was obtained by using either burner. Hence, the method is not acceptable for the determination of cadmium.

Table 12. Summary of Data on Aqueous Lead

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.200 mg/liter								
P.E., 303, Premix	3	0	0.2793	+0.079	0.1119	39.67	40.07	1.1098
P.E., 303, Boling	7	0	0.1720	-0.028	0.0516	14.00	29.98	0.2056
J.A., (82-500), Hetco	3	0	0.1507	-0.049	0.1315	24.67	87.25	1.3035
Overall	19	2	0.1892	-0.011	0.0657	5.41	34.76	0.1879
Sample 2, 0.100 mg/liter								
P.E., 303, Premix	2	1	0.0970	-0.003	0.0000	3.00	0.00	0.0000
P.E., 303, Boling	7	0	0.0840	-0.016	0.0566	16.00	67.33	0.2266
J.A., (82-500), Hetco	3	0	0.1040	+0.004	0.0963	4.00	92.57	0.9546
Overall	19	0	0.1082	+0.008	0.0667	8.16	61.65	0.1856
Sample 3, 0.050 mg/liter								
P.E., 303, Premix	3	0	0.0517	+0.002	0.0015	3.33	2.96	0.0151
P.E., 303, Boling	7	0	0.0476	-0.002	0.0364	4.86	76.48	0.1458
J.A., (82-500), Hetco	3	0	0.0767	+0.027	0.0802	53.33	104.62	0.7953
Overall	19	0	0.0641	+0.014	0.0452	28.21	70.55	0.1259

Table 13. Summary of Data on Extracted Lead

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.200 mg/liter								
P.E., 303, Premix	9	0	0.2123	+0.012	0.0456	6.17	21.49	0.1611
P.E., 303, Boling	8	0	0.1835	-0.017	0.0883	8.25	48.10	0.3294
Overall	24	0	0.2062	+0.006	0.0654	3.08	31.72	0.1734
Sample 2, 0.100 mg/liter								
P.E., 303, Premix	9	1	0.1051	+0.005	0.0228	5.13	21.67	0.0850
P.E., 303, Boling	8	0	0.0955	-0.005	0.0527	4.50	55.22	0.1968
Overall	24	1	0.1031	+0.003	0.0338	3.09	32.80	0.0904
Sample 3, 0.050 mg/liter								
P.E., 303, Premix	9	1	0.0595	+0.010	0.0140	19.00	23.46	0.0521
P.E., 303, Boling	8	1	0.0516	+0.002	0.0275	3.14	53.33	0.1102
Overall	24	3	0.0591	+0.009	0.0158	18.19	26.65	0.0429

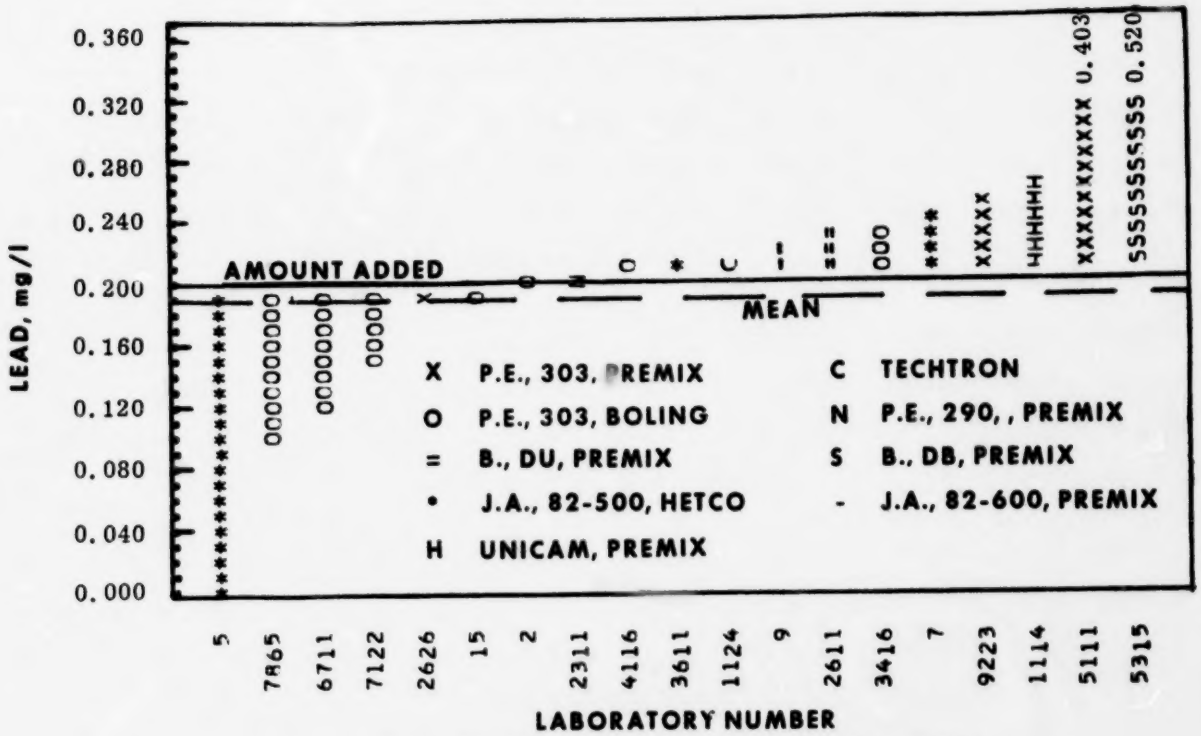

Figure 28. Bar graph for aqueous lead in sample 1.

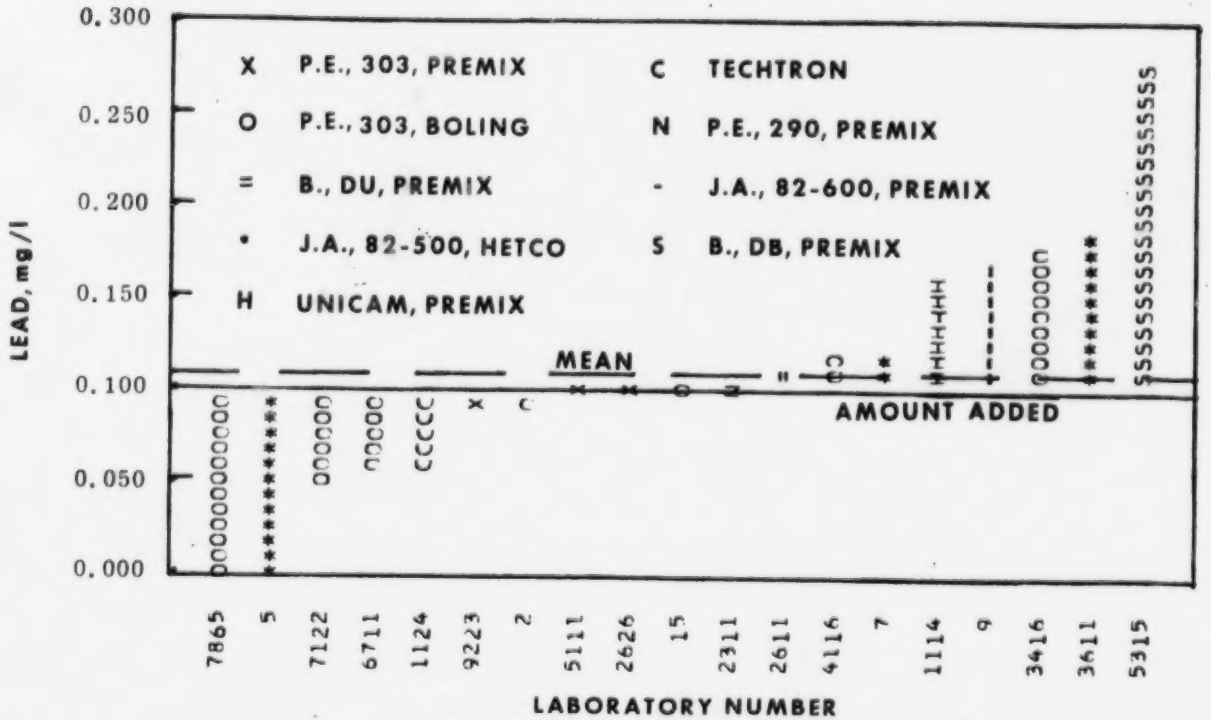

Figure 29. Bar graph for aqueous lead in sample 2.

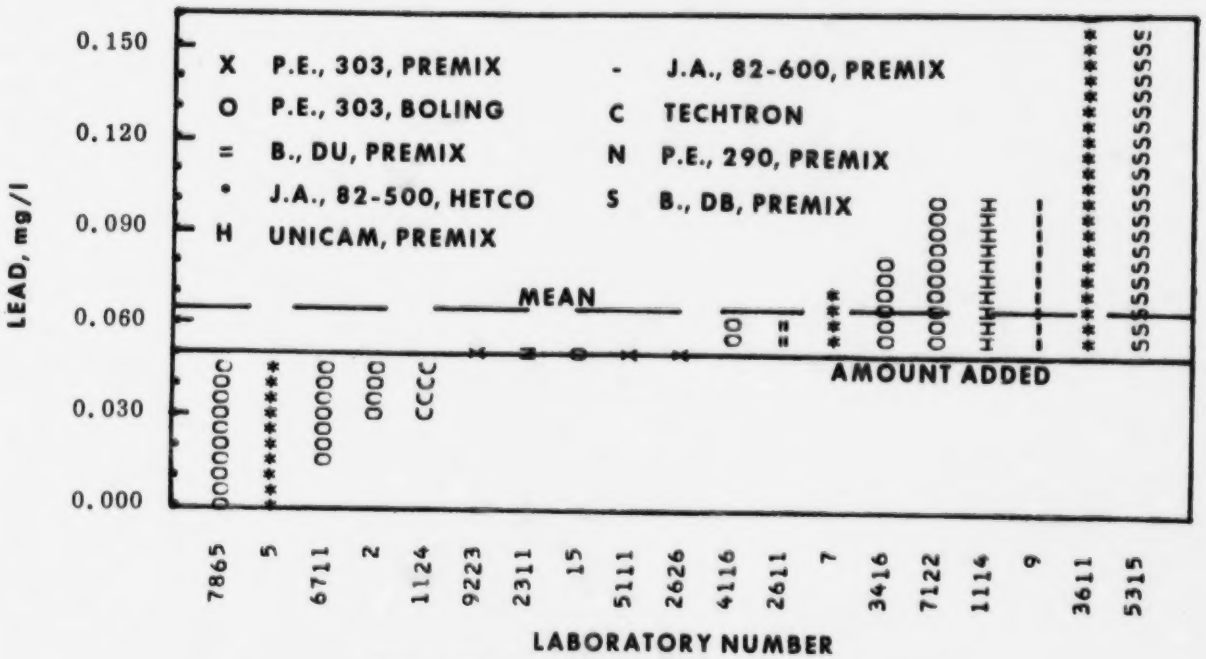

Figure 30. Bar graph for aqueous lead in sample 3.

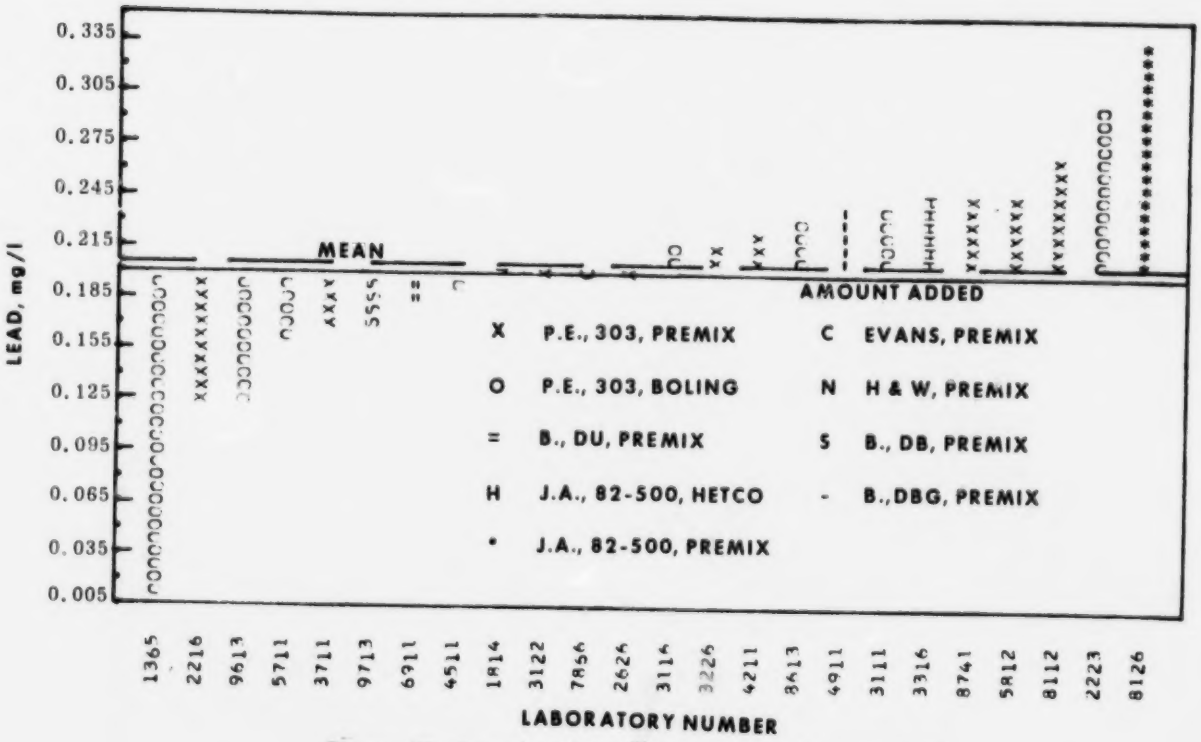

Figure 31. Bar graph for extracted lead in sample 1.

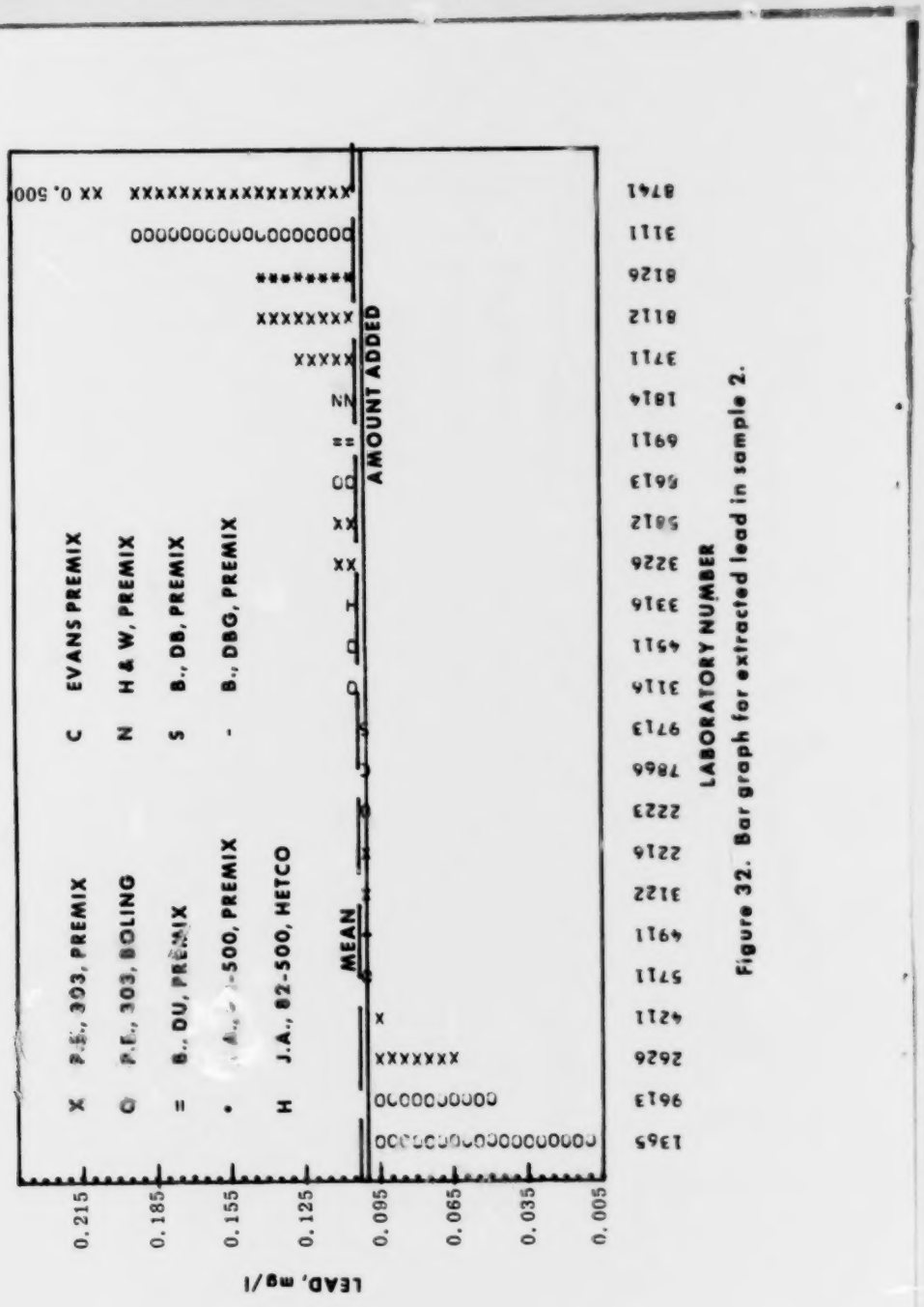

Figure 32. Bar graph for extracted lead in sample 2.

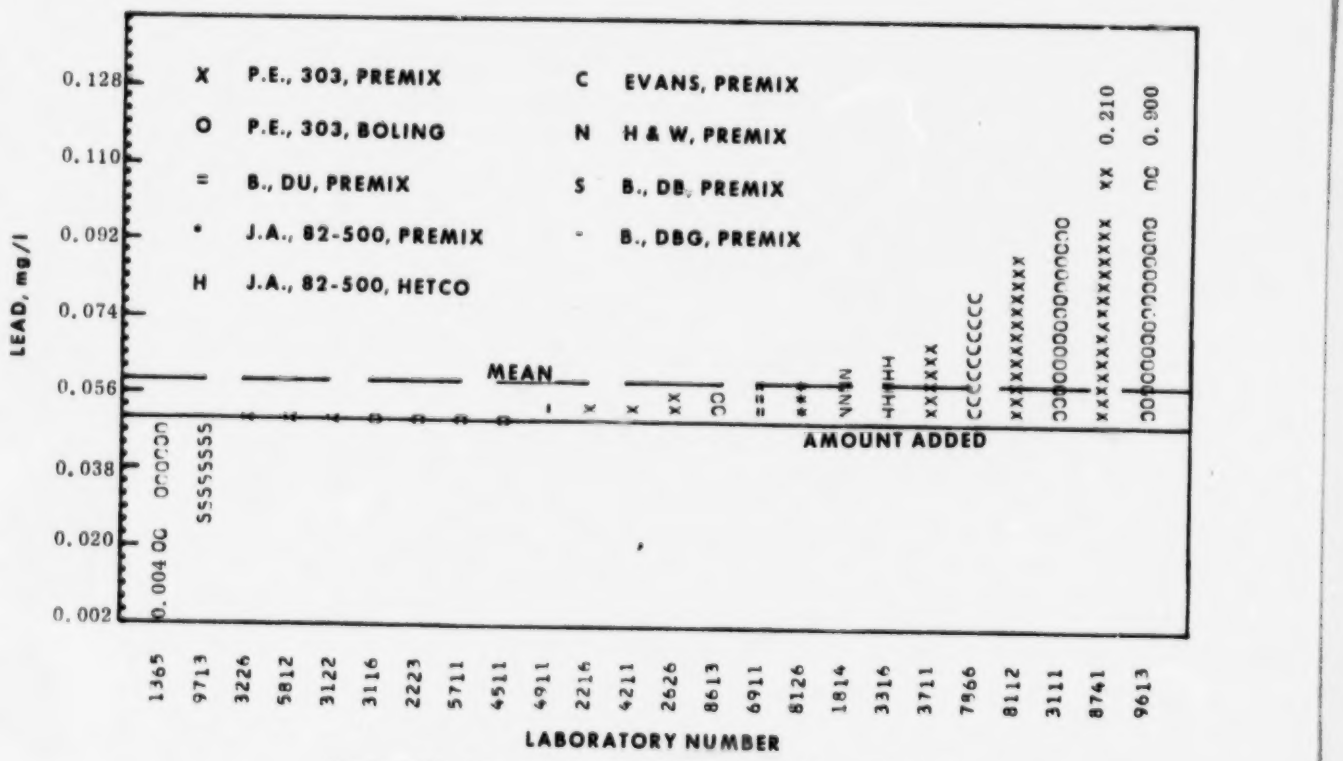

Figure 33. Bar graph for extracted lead in sample 3.

Table 14. Summary of Data on Aqueous Cadmium

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.010 mg/liter					
P.E., 303, Premix	5	0	0.0168	+0.007	0.0113	68.00	67.53	0.0576
P.E., 303, Boling	8	0	0.0128	+0.003	0.0068	27.50	53.32	0.0254
J.A., (82-500), Hetco	3	0	0.0107	+0.001	0.0101	6.67	94.37	0.0998
B., DB, Premix	3	0	0.0220	+0.012	0.0122	120.00	55.30	0.1206
Overall	25	0	0.0146	+0.005	0.0086	45.60	59.28	0.0227
			Sample 2, 0.050 mg/liter					
P.E., 303, Premix	5	0	0.0608	+0.011	0.0143	21.60	23.47	0.0725
P.E., 303, Boling	8	1	0.0510	+0.001	0.0040	2.00	7.77	0.0148
J.A., (82-500), Hetco	3	0	0.0483	-0.002	0.0076	3.33	15.80	0.0757
B., DB, Premix	3	0	0.0623	+0.012	0.0178	24.67	28.53	0.1764
Overall	26	0	0.0541	+0.004	0.0117	8.15	21.62	0.0305
			Sample 3, 0.100 mg/liter					
P.E., 303, Premix	5	0	0.1168	+0.017	0.0195	16.80	16.66	0.0988
P.E., 303, Boling	9	0	0.1034	+0.003	0.0080	3.44	7.74	0.0283
J.A., (82-500), Hetco	3	0	0.1000	0.000	0.0000	0.00	0.00	0.0000
B., DB, Premix	3	0	0.1127	+0.013	0.0258	12.67	22.91	0.2560
Overall	26	0	0.1075	+0.008	0.0149	7.50	13.84	0.0388

Table 15. Summary of Data on Extracted Cadmium

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.010 mg/liter								
P.E., 303, Premix	5	0	0.0132	+0.003	0.0102	32.00	77.52	0.0520
P.E., 303, Boling	7	1	0.0100	0.000	0.0061	0.00	60.99	0.0269
Overall	13	1	0.0108	+0.001	0.0079	8.33	72.77	0.0249
Sample 2, 0.050 mg/liter								
P.E., 303, Premix	5	0	0.0428	-0.007	0.0205	14.40	47.95	0.1042
P.E., 303, Boling	7	2	0.0494	-0.001	0.0048	1.20	9.77	0.0245
Overall	13	1	0.0433	-0.007	0.0190	13.33	43.84	0.0601
Sample 3, 0.100 mg/liter								
P.E., 303, Premix	5	0	0.0686	-0.031	0.0479	31.40	69.82	0.2433
P.E., 303, Boling	7	1	0.0850	-0.015	0.0445	15.00	52.39	0.1965
Overall	13	1	0.0820	-0.018	0.0452	18.00	55.08	0.1428

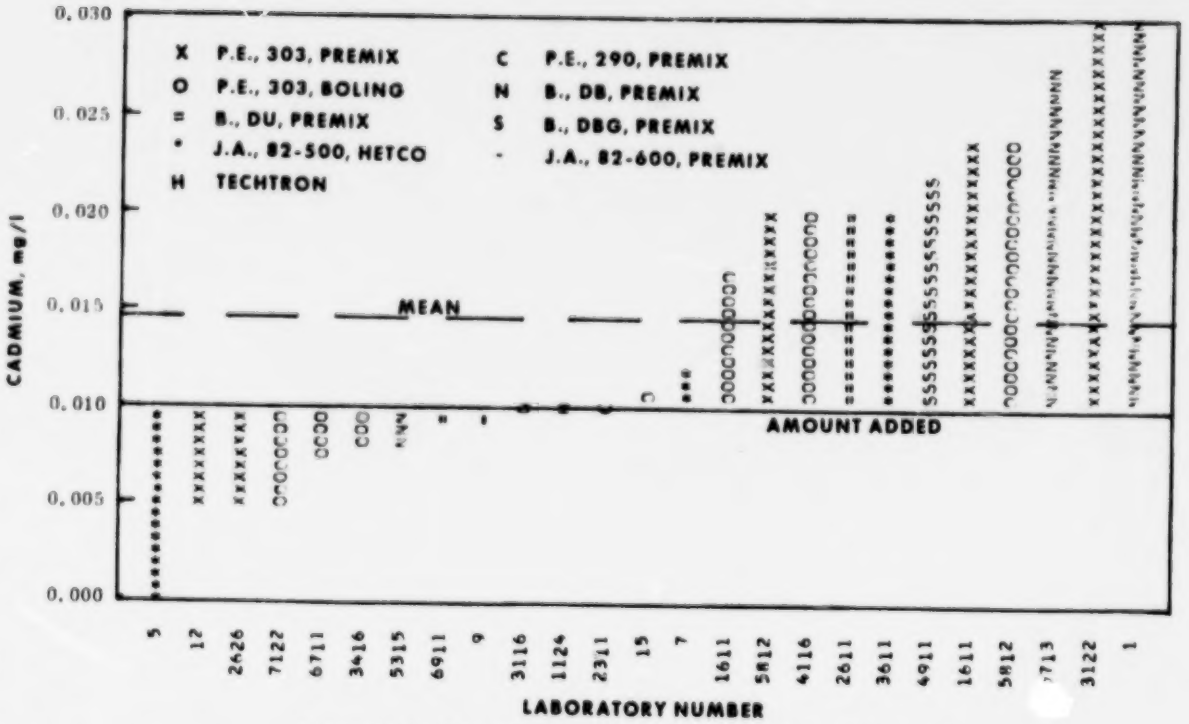

Figure 34. Bar graph for aqueous cadmium in sample 1.

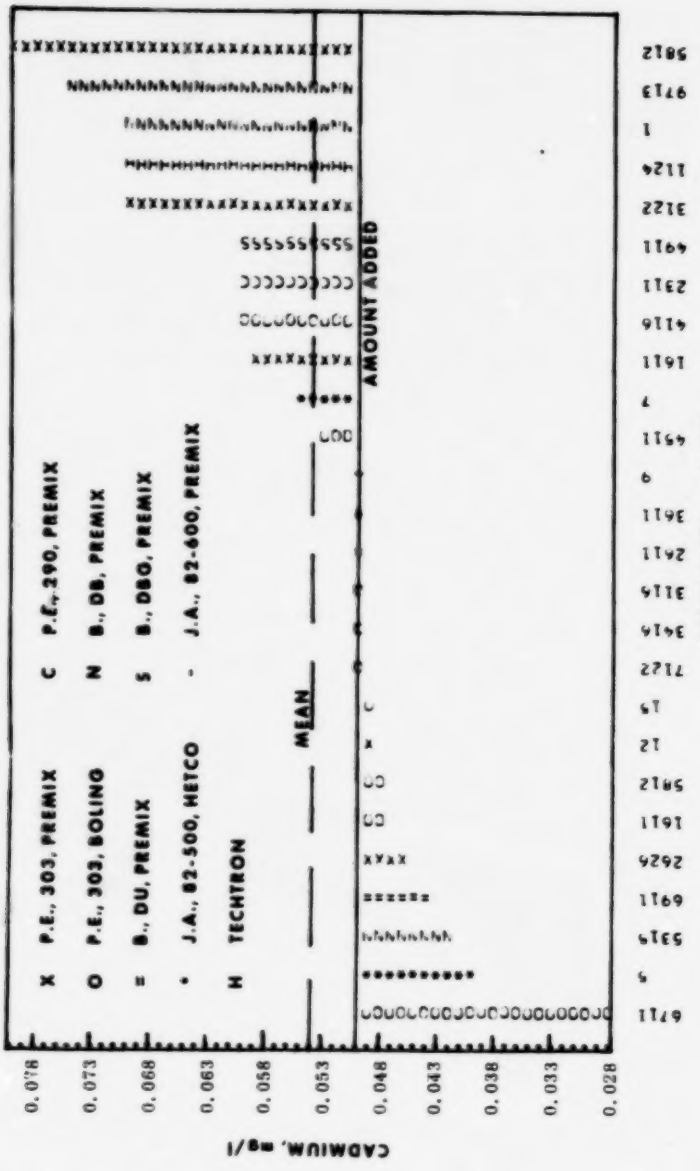

Figure 35. Bar graph for aqueous cadmium in sample 2.

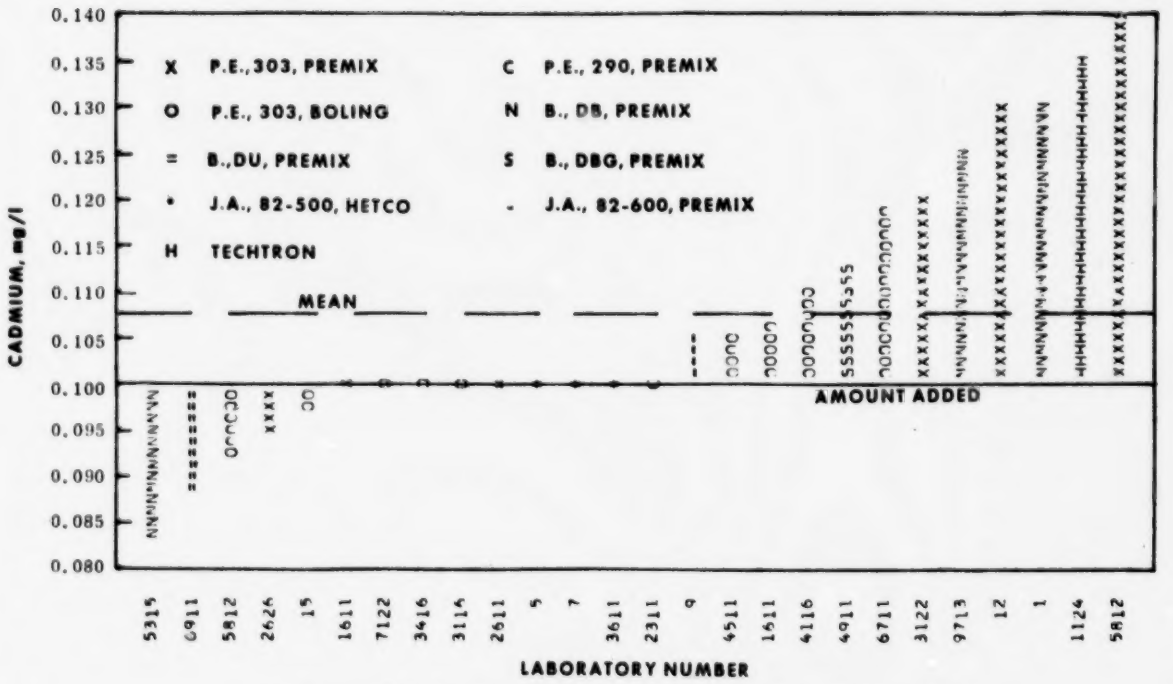

Figure 36. Bar graph for aqueous cadmium in sample 3.

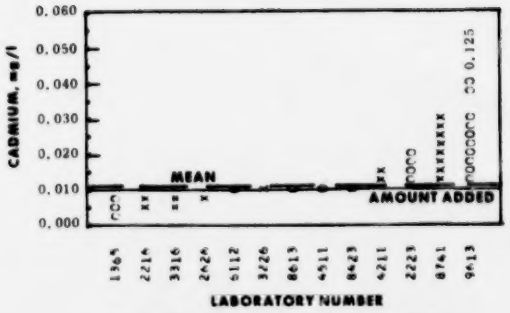

Figure 37. Bar graph for extracted cadmium in sample 1.

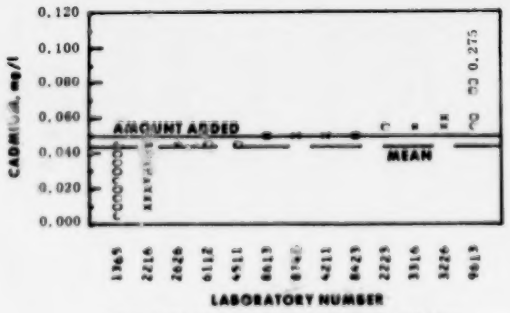

Figure 38. Bar graph for extracted cadmium in sample 2.

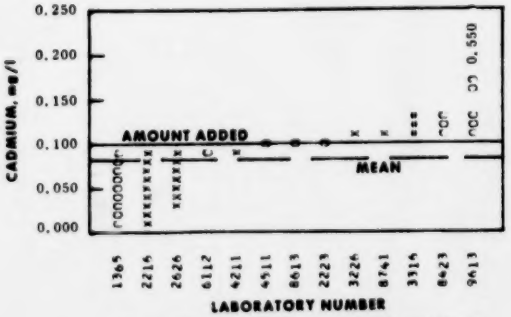

Figure 39. Bar graph for extracted cadmium in sample 3.

X P.E., 303, PREMIX

O P.E., 303, BOLING

J.A., 82-600, HETCO

IRON (Tables 16 and 17, Figures 40, 41, 42, 43, 44 and 45)

In the determination of iron by direct aspiration of the aqueous sample into the burner (Table 16, Figures 40, 41 and 42), 12 laboratories analyzed the samples by using a Perkin-Elmer spectrophotometer equipped with a premix burner, and 19 analyzed the samples by using an identical instrument equipped with a Boling burner. In the analysis of sample 1 (0.100 mg/liter), the accuracy obtained by using either burner was acceptable, but the precision of both was poor. There was no significant statistical difference in either precision or accuracy. In the analysis of sample 2 (0.500 mg/liter), there was no significant difference in the results obtained by either burner, and both the precision (6.16 vs. 5.24) and the accuracy (1.14 vs. 5.02) were excellent. In the analysis of sample 3 (0.300 mg/liter), similar results were obtained. Since the drinking water standard is set at this level, this is obviously an acceptable method for the determination of iron, regardless of which type of burner or instrument is used as indicated by an overall precision of 16.53 and accuracy of 0.64 percent at the 0.300 mg/liter level.

Too few laboratories submitted results in the determination of iron by chelation and extraction before aspirating into the flame (Table 17, Figures 43, 44 and 45) to make possible any statistically significant comparison. In general, however, it would appear that both the precision and accuracy are unacceptable, regardless of the burner or instrument used.

DISCUSSION

In this study, all participants were instructed to use and hence, presumably followed, the provided procedure. Nevertheless, the participant actually had to choose whether to analyze the sample by aspirating it directly into the burner or to concentrate it by chelation with ammonium pyrrolidine dithiocarbamate and extraction into methyl isobutyl ketone before aspiration into the flame. The choice, theoretically, depended upon the sensitivity of the method in determining that particular metal. As a result, some participants did it one way and others, the opposite way.

Since the type of instrument used for analysis, obviously, could not be controlled, the participant was also given a choice of instrument (manufacturer) and burner type to be used.

In analyzing the results obtained in this study, an attempt was made to acquire information on the precision and accuracy of each of the variations used by the participants. Unfortunately, this was not always possible because many of the results were from either a single or an average of

Table 16. Summary of Data on Aqueous Iron

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
			Sample 1, 0.100 mg/liter					
P.E., 303, Premix	12	0	0.0920	-0.008	0.0308	8.00	33.48	0.1041
P.E., 303, Boling	19	0	0.0947	-0.005	0.0320	5.26	33.76	0.0890
J.A., (82-500), Hetco	3	0	0.1423	+0.042	0.0513	42.33	36.06	0.5089
B., DBG, Premix	2	1	0.1000	------	------	-----	-----	------
Overall	46	1	0.1156	+0.016	0.0527	15.60	45.59	0.1269
			Sample 2, 0.500 mg/liter					
P.E., 303, Premix	12	2	0.5251	+0.025	0.0275	5.02	5.24	0.0929
P.E., 303, Boling	19	2	0.5057	+0.006	0.0312	1.14	6.16	0.0890
J.A., (82-500), Hetco	3	0	0.5977	+0.098	0.1433	19.53	23.98	1.4210
B., DBG, Premix	3	0	0.4893	-0.011	0.0393	2.13	8.04	0.3899
Overall	46	5	0.5203	+0.020	0.0480	4.06	9.22	0.1170
			Sample 3, 0.300 mg/liter					
P.E., 303, Premix	12	2	0.3058	+0.006	0.0468	1.93	15.30	0.1581
P.E., 303, Boling	19	0	0.2851	-0.015	0.0502	4.98	17.62	0.1398
J.A., (82-500), Hetco	3	0	0.3407	+0.041	0.0820	13.56	24.08	0.8134
B., DBG, Premix	3	0	0.2957	-0.004	0.0093	1.44	3.14	0.0921
Overall	46	3	0.3019	+0.002	0.0499	0.64	16.53	0.1209

Table 17. Summary of Data on Extracted Iron

Instrumentation	No. of Results	No. of Outliers	Mean	Mean Error	Std. Dev.	Relative Error	Relative Std. Dev.	95% Tol. Limits
Sample 1, 0.100 mg/liter								
Overall	6	0	0.1388	+0.039	0.1159	38.83	83.48	0.5116
Sample 2, 0.500 mg/liter								
Overall	6	0	0.3003	-0.200		39.93	68.56	0.9089
Sample 3, 0.300 mg/liter								
Overall	6	0	0.2940	-0.006	0.2073	2.00	70.52	0.9151

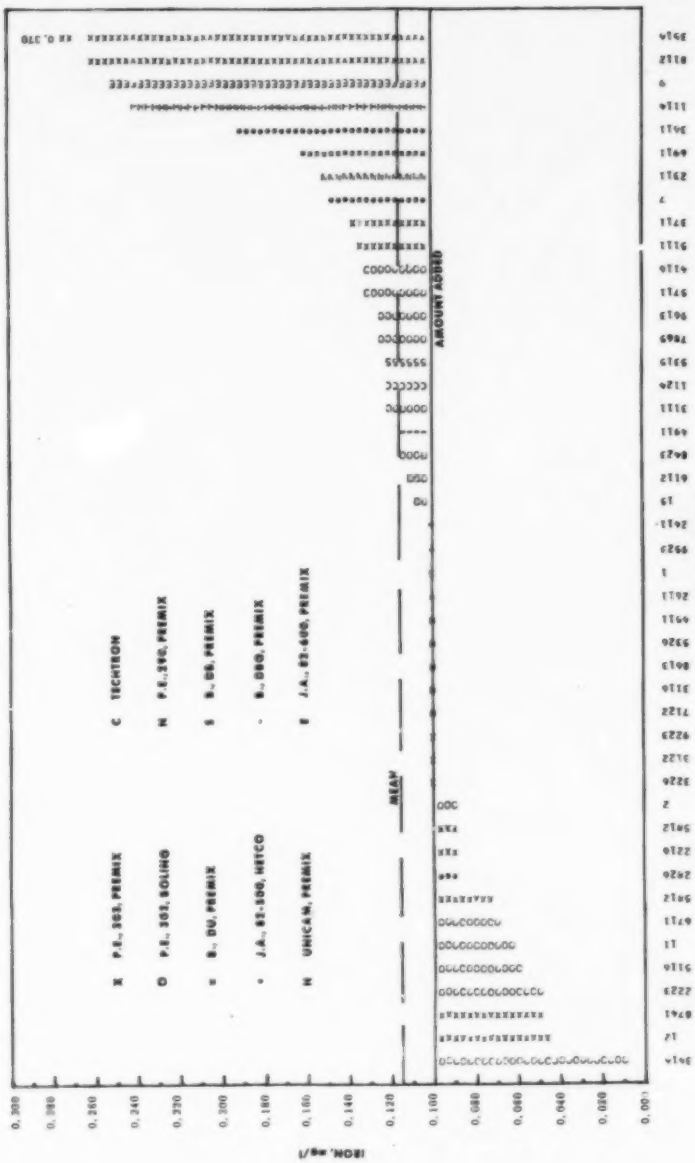

Figure 40. Bar graph for aqueous iron in sample 1.

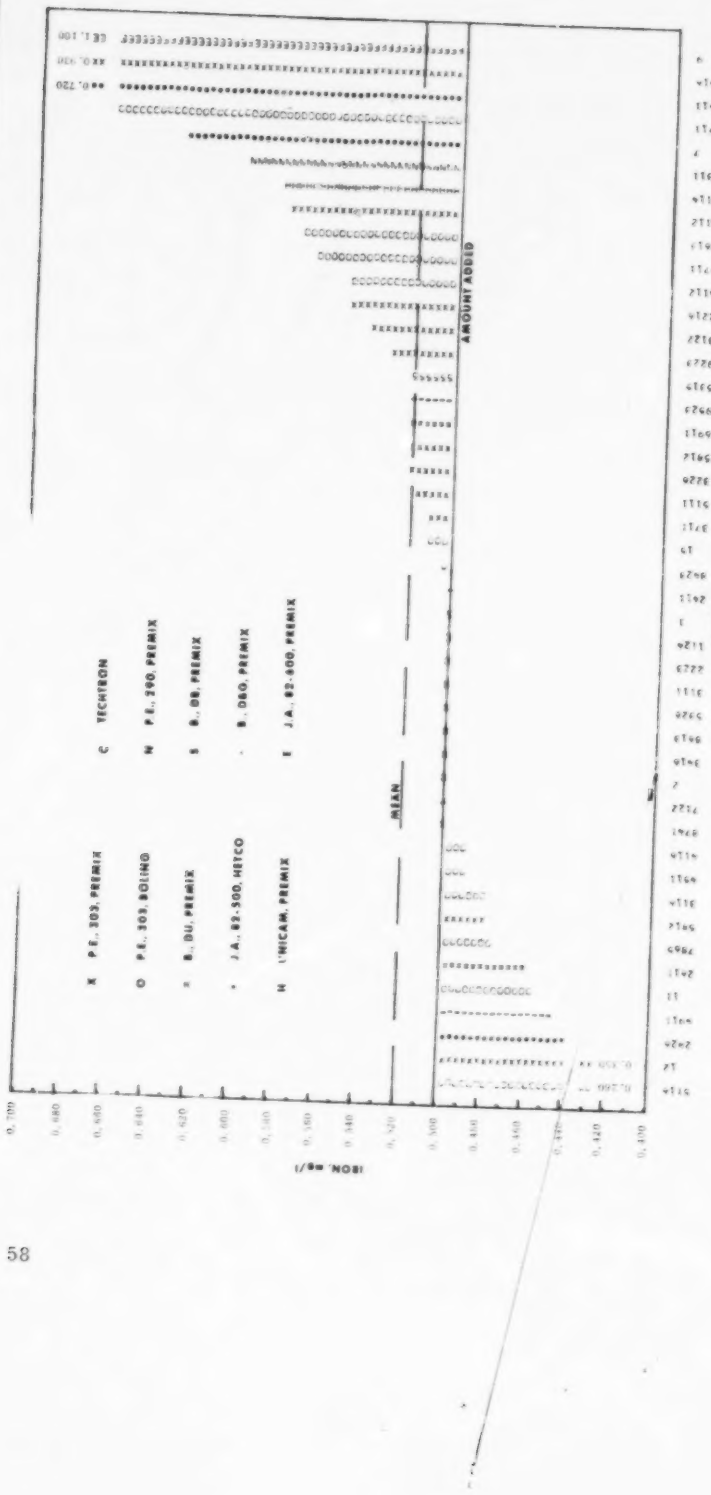

Figure 41. Bar graph for aqueous iron in sample 2.

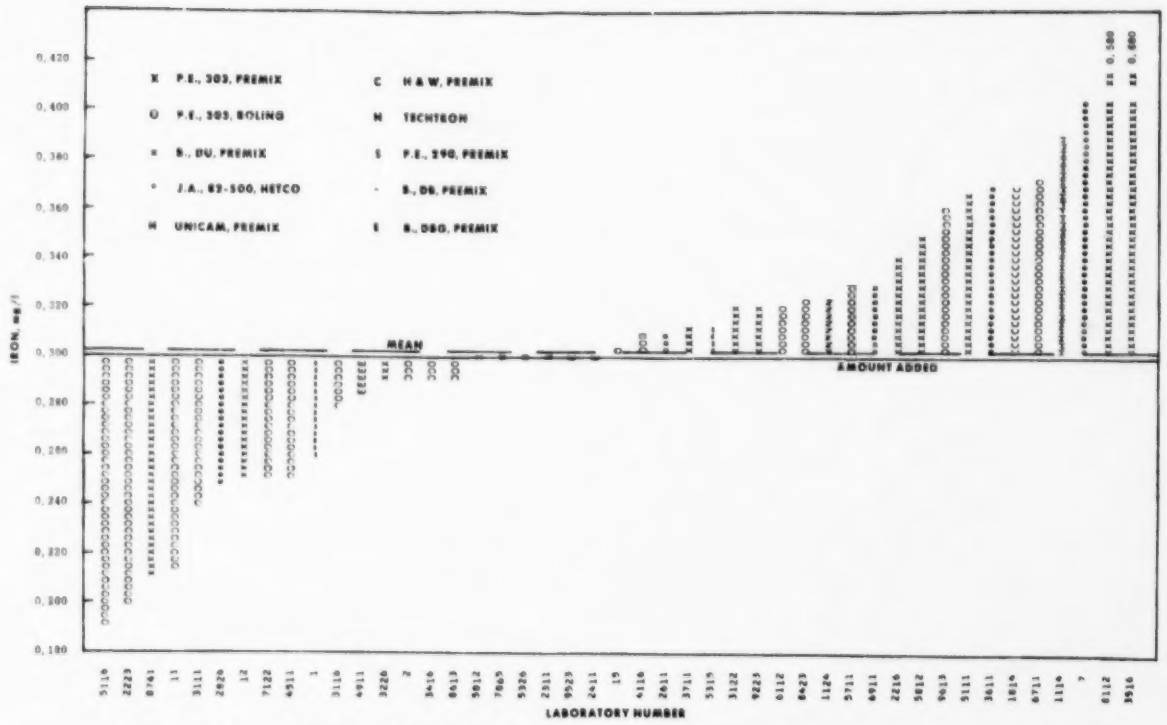

Figure 42. Bar graph for aqueous iron in sample 3.

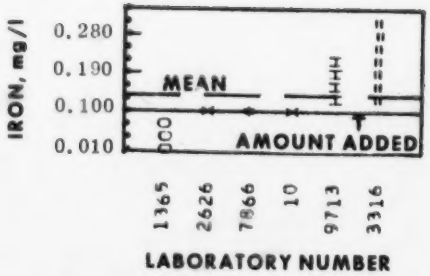

Figure 43. Bar graph for extracted iron in sample 1.

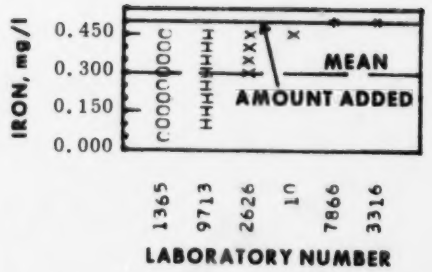

Figure 44. Bar graph for extracted iron in sample 2.

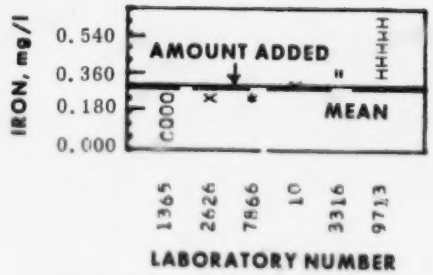

Figure 45. Bar graph for extracted iron in sample 3.

X P.E., 303, PREMIX

O P.E., 303, PREMIX

= J.A., 82-500, HETCO

two to five determinations obtained by using a particular instrument, burner, or extraction procedure, and, hence, not represented by sufficient number of determinations to be statistically significant.

In order to ascertain whether a particular metal could be determined satisfactorily by atomic absorption spectrophotometry and whether that method for the metal should be recommended for inclusion in Standard Methods for the Examination of Water and Wastewater, the results have been evaluated in terms of whether the sensitivity of the method is sufficient to permit the measurement of the metal at the concentration set for drinking water standards, with a reasonable degree of precision and accuracy.

COMMENTS OF THE PARTICIPANTS

EXTRACTION OF MANGANESE

Seven participants commented that manganese does not extract with methyl isobutyl ketone after complexing with ammonium pyrrolidine dithiocarbamate under the pH conditions given. One participant suggested that manganese could be extracted better at a pH above 4.5. Another commented that the inefficient extraction of manganese with the procedure given corresponded to the observations of Mansel, who noted that at least 2.25 ml of 5 percent ammonium pyrrolidine dithiocarbamate was required to extract manganese completely from only 45 ml of water with 10 ml of methyl isobutyl ketone at a pH 3.0.

SOLUBILITY OF APDC

Three participants commented on the solubility of ammonium pyrrolidine dithiocarbamate. One participant asked whether we had any information on the shelf life and stated that the insoluble fraction of K and K APDC had apparently lost its ammonia group and could be removed by filtration without impairing the reagent, except that, I suppose, its chelating capacity had been considerably reduced. Another reported that the product obtained from K and K was insoluble, whereas the product ordered from Eastman Kodak was soluble.

METHYL ISOBUTYL KETONE EXTRACTION

Four participants commented upon the methyl isobutyl ketone extraction procedure, suggesting the need for improvement. In general, the problem was the solubility of MIBK. It was suggested that larger volumes be used in the first extraction or another solvent of lower solubility be used. Another participant suggested that a heavier-than-water solvent would be more satisfactory in that the less dense solvents present a real manipulation problem.

ACIDIFICATION WITH HYDROCHLORIC ACID

Two participants commented upon the acidification with hydrochloric acid. One participant stated that acidification with hydrochloric acid produced erratic results and that the use of nitric acid solved the problem. The other stated that he felt the use of hydrochloric acid was a mistake since a significant concentration of silver would be precipitated.

pH OF OXIDATION

One participant commented that the pH of the sample and standards before the addition of permanganate should be adjusted to a pH of 2 to 3 in order to visually observe the disappearance of excess permanganate when adding the azide in the oxidation of chromium. Another commented that hydrochloric acid interferes with the oxidation step when $KMnO_4$ is used, and that the procedure is satisfactory if the hydrochloric acid is omitted; however, the solution cannot be basic.

MISCELLANEOUS

In the manganese determination, if lanthanum instead of calcium is added to remove the silica interference, the same aliquot can be used to determine both manganese and calcium.

SUMMARY AND CONCLUSIONS

In the determination of zinc, copper, magnesium, and iron, the method of direct aspiration of the aqueous sample into the burner was found to be acceptable regardless of the type of instrument or burner used. The same method was also found acceptable for the determination of chromium, manganese, and silver, provided a Boling rather than a premix burner was used. In fact, in most cases, significantly more precise results were obtained for all the metals by using a Boling burner. The only exception was in the determination of lead. In this case, however, the method of direct aspiration of the aqueous sample into the burner was unacceptable. Barely acceptable results (accuracy at the 0.050 mg/l level was 19.0 percent and the precision was 23.46 percent) were obtained for lead by chelation and extraction of the sample before aspiration into a premix burner flame. In the determination of cadmium, neither procedure was acceptable (low sensitivity), although both accuracy and precision were better with the Boling burner.

APPENDICES

APPENDIX A.

METHOD OF ANALYSIS FOR METALS IN NATURAL AND TREATED WATERS IN THE ABSENCE OF GROSS POLLUTION BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

Magnesium, copper, and zinc can be determined by atomic absorption with a sensitivity limit that is lower than the established drinking water standards. Therefore, in evaluating the safety of drinking water, these metals can be determined by simply aspirating the aqueous solution into the flame. Iron, in all probability, can likewise be determined, although the precision and accuracy may not be as good. The determination of manganese, chromium, cadmium, lead, and silver at the concentration set for drinking water may be difficult. At the lower concentrations, however, these metals can probably be determined by complexing with a chelating compound, extracting the chelate into an organic solvent, and aspirating the organic phase directly into the burner.

1. GENERAL DISCUSSION

1.1. Principle: Atomic absorption resembles emission flame photometry in that a sample is aspirated into a flame and atomized. The major difference lies in the fact that flame photometry measures the amount of light emitted, whereas in atomic absorption spectroscopy a light beam is directed through the flame, into a monochromator, and onto a detector which measures the amount of light absorbed. Absorption is more sensitive because it depends upon the presence of free unexcited atoms, and in the usual flames the ratio of unexcited to excited atoms at a given moment is very high. Since each metallic element has its own characteristic absorption wave length, a source lamp composed of that element is employed for each element, and hence the method is relatively free of spectral or radiation interferences. The amount absorbed in the flame is proportional to the concentration of the element in the sample.

1.2. Interference: The most troublesome type of interference is usually termed "chemical," and results from the lack of absorption from atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule (in the case of phosphate interference with magnesium) or because the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The interference of phosphate in the magnesium determination can be overcome by the addition of lanthanum. In the determination of manganese, the addition of calcium similarly eliminates the silica interference.

2. APPARATUS

2.1. Atomic absorption spectrophotometer.

2.2. Autoclave.

2.3. Glassware: All glassware, including sample bottles, should be rinsed with 1 + 1 HNO_3 followed by deionized distilled water to avoid contamination errors.

3. REAGENTS

3.1. Deionized distilled water: Prepare by passing distilled water through a mixed bed of ion-exchange resins. Use de-ionized distilled water for the preparation of all reagents, calibration standards, and as dilution water.

3.2. Hydrochloric acid, concentrated.

3.3. Stock metal solution: Prepare as directed in Table I.

3.4. Standard solutions: In general, prepare a series of standard solutions containing 0.01, 0.02, 0.04, 0.06, 0.08, and 0.10 mg/l by appropriate dilution of the stock solution.

3.5. Fuel and oxidant: Use an air-acetylene flame for all the metals in Table I.

3.6. Special reagent for the determination of magnesium:
Stock lanthanum solution: Dissolve 58.65 g lanthanum oxide, La_2O_3, in 250 ml of concentrated HCl. Dilute to 1000 ml with distilled water to produce a solution containing 5 g La per 100 ml. Add the acid slowly until the material is dissolved. Add sufficient amounts of this solution to the magnesium standards and unknowns in order to obtain a final working concentration of 1 g La per 100 ml volume.

3.7. Special reagent for the determination of manganese:
Stock calcium solution: Dissolve 2.4752 g calcium carbonate in the minimum volume of hydrochloric acid, and dilute to one liter to obtain a solution containing 1000 mg/l calcium. Add sufficient amounts of this solution to manganese standards and unknowns to produce a final working concentration of 50 mg/l.

3.8. Special reagents for the determination of low concentrations of lead, cadmium, chromium, manganese, and silver:

a. Methyl isobutyl ketone: Check for contamination by burning in the flame. If contaminated, wash one volume of methyl isobutyl ketone

with five volumes of 1 + 99 hydrochloric acid solution. Remove the excess acid by washing with de-ionized distilled water.

b. Ammonium pyrrolidine dithiocarbamate solution: Dissolve 4 g in 100 ml distilled water (K and K Laboratories, Inc., Plainview, N. Y.).

Table I

Metal	Wave length mμ	Stock solution page reference[a]	Sensitivity (ppm per 1%)[b]
Zinc	213.8	318	0.04
Chromium	357.9	124	0.15
Cadmium	228.8	69	0.04
Copper	324.7	131	0.20
Magnesium[c]	285.2	171	0.015
Manganese[d]	279.4	174	0.15
Silver	328.1	268	0.10
Lead	283.3	164	0.50
Iron	248.3	157	0.30

[a] SMEWW, 12th Ed.

[b] ppm of metal for 1% absorption in an aqueous solution.

[c] Add lanthanum to overcome phosphate interference.

[d] Add calcium to overcome silica interference.

4. PROCEDURE

4.1. Pretreatment of sample: Acidify all water samples with hydrochloric acid (1 ml concentrated hydrochloric acid per 100 ml sample), and autoclave at 250° F for 1 hour to solubilize particulate matter.

4.2. Precautions: Install the spectrophotometer on a level bench or table, with the burner under a vent.

4.3. Instrument operation: Because the differences between the makes and models of satisfactory atomic absorption spectrophotometers make impossible the formulation of detailed instructions applicable to every instrument, follow the manufacturer's operating instructions. As a rule, choose the correct hollow cathode lamp, install, and align in the instrument; position the monochromator at the correct wave length (Table I); select the proper monochromator slit width; set the light source current according to the manufacturer's recommendation; light the flame and regulate the flow of fuel and oxidant; adjust the burner

for maximum percent absorption and stability; and balance the photometer. Then, run the standards which are needed for the working curves, and plot the concentrations of the standards against absorbance. If necessary on some instruments, convert percent absorption to absorbance. The calibration curves are nearly linear for most elements. Next run the samples and read the concentration of each sample from the calibration curve. For best results run standards each time a sample or series of samples is run.

4.4. Special procedural steps for low concentrations of lead, cadmium chromium, manganese, and silver: Acidify 200 ml water sample to a pH of 2.2 to 2.8 by adding hydrochloric acid. Add 1 ml ammonium pyrroli line dithiocarbamate solution and 5 ml methyl isobutyl ketone, and shake for 2 minutes manually. If an emulsion forms at the interface of the two layers, centrifuge for 10 minutes. Repeat the extraction with a second 5 ml of methyl isobutyl ketone, and combine the two extracts. Determine metal in the organic phase against standards which have similarly been extracted. If a precipitate forms in the solvent phase during the first extraction, add an additional 5 ml of methyl isobutyl ketone, and repeat the extraction with a second 10 ml. This, of course, will reduce the concentration factor and sensitivity of the method and should not be done unless necessary.

4.5. Aspiration of an organic solvent into the flame: When working with organic solvents, the fuel-to-air ratio must be reduced as the burning of the organic solvent contributes to the fuel supply and, in addition to lifting the flame off the burner, may produce an undesirable luminescent flame. In setting the fuel-to-air ratio of the gas mixture at the burner, start with the settings recommended by the manufacturer for the analysis and gradually reduce the fuel flow (while the solvent is being aspirated) until the flame is as blue as possible and does not lift off the burner. Generally, the fuel flow can be cut almost in half.

4.6. Oxidation of trivalent chromium to the hexavalent form: Trivalent chromium apparently does not complex with ammonium pyrrolidine dithiocarbamate and, hence, cannot be extracted into methyl isobutyl ketone. It is, therefore, necessary to oxidize the trivalent chromium to the hexavalent form before undertaking analysis by this technique. To oxidize the chromium, bring the sample to a boil, and add sufficient $KMnO_4$ (0.1N, dissolve 3.16 g $KMnO_4$ in distilled water and dilute to 1000 ml) dropwise so that a faint pink color persists as the solution continues to boil for 10 minutes. Then, add NaN_3 (0.5 g NaN_3 in distilled water diluted to 100 ml) solution dropwise and continue boiling until the solution becomes colorless. Boil for about 2 min. between azide additions to guard against the use of excess azide. Cool the sample.

5. CALCULATION

5.1. For aqueous sample, refer directly to the calibration curve

$$\text{mg/l} = (\text{mg/l in aliquot}) \times D$$

$$D = \text{dilution ratio} = \frac{\text{ml sample} + \text{ml water} + 1\ (\text{ml acid})}{\text{ml sample}}$$

5.2. For extracted sample, refer to the extracted standard calibration curve.

$$\text{mg/l} = (\text{mg/l in extract}) \times C$$

$$C = \text{concentration ratio} = \frac{\text{ml solvent}}{\text{ml sample}}$$

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APPENDIX B.
TABULATION OF RESULTS

Table B-1. Tabulation of Results for Zinc

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.120	11	3116	0.060	2
2	0.060	2	3122	0.050	1
5	0.040	5	3226	0.600	1
6	0.140	2	3316	0.037	5
7	0.054	5	3416	0.019	2
9	0.075	13	3516	0.065	1
11	0.048	2	3611	0.060	5
12	0.090	1	3711	0.075	1
15	0.049	2	4116	0.040	2
1114	0.090	6	4211	0.030	2
1124	0.050	9	4511	0.050	2
1365	0.051	2	4911	0.080	12
1611	0.083	2	5111	0.048	1
1611	0.040	1	5315	0.051	11
1814	0.050	8	5326	0.050	2
2216	0.060	1	5711	0.202	2
2223	0.050	2	5812	0.100	1
2311	0.050	10	5812	0.050	1
2411	0.056	12	6112	0.030	2
2611	0.180	3	6711	0.028	2
2626	0.052	1	6911	0.050	3
2826	0.070	5	7122	0.005	2
3111	0.060	2	7865	0.000	2

(Table B-1 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
7866	0.060	7	9223	0.060	1
8112	0.095	1	9523	0.055	12
8126	1.000	4	9613	0.070	2
8423	0.050	2	9713	0.005	11
8613	0.049	2	9914	0.070	2
8741	0.200	1			
		Sample 2, 0.500 mg/liter			
1	0.650	11	2411	0.565	12
2	0.520	2	2611	0.680	3
5	0.480	5	2626	0.460	1
6	0.100	2	2826	0.550	5
7	0.429	5	3111	0.550	2
11	0.503	2	3116	0.490	2
12	0.520	1	3122	0.430	1
15	0.500	2	3226	0.500	1
1114	0.735	6	3316	0.500	5
1124	0.465	9	3416	0.500	2
1365	0.505	2	3516	0.507	1
1611	0.520	2	3611	0.420	5
1611	0.498	1	3711	0.540	1
1814	0.600	8	4116	0.540	2
2216	0.530	1	4211	0.500	2
2223	0.510	2	4511	0.500	2
2311	0.700	10	4911	0.490	12

(Table B-1 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
5111	0.498	1	7866	0.570	7
5315	0.520	11	8112	0.550	1
5326	0.500	2	8126	0.400	4
5711	0.248	2	8423	0.500	2
5812	0.550	1	8613	0.500	2
5812	0.510	1	8741	0.520	1
6112	0.500	2	9223	0.548	1
6711	0.491	2	9523	0.455	12
6911	0.440	3	9613	0.500	2
7122	0.050	2	9713	0.250	11
7865	0.425	2	9914	0.500	2
		Sample 3, 1.000 mg/liter			
1611	1.040	2	1	1.780	11
1814	1.200	8	2	1.050	2
2216	0.940	1	5	0.960	5
2223	1.020	2	6	0.040	2
2311	1.300	10	7	0.879	5
2411	1.250	12	11	0.990	2
2611	2.400	3	12	0.990	1
2626	1.000	1	15	1.000	2
2826	1.100	5	1114	1.105	6
3111	1.170	2	1124	1.360	9
3116	0.990	2	1365	0.616	2
3122	0.910	1	1611	0.948	1

(Table B-1 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
3226	0.980	1	6112	1.000	2
3316	1.013	5	6711	0.942	2
3416	0.940	2	6911	0.880	3
3516	0.984	1	7122	0.100	2
3611	0.890	5	7865	0.925	2
3711	1.020	1	7866	0.970	7
4116	0.980	2	8112	1.125	1
4211	0.973	2	8126	0.100	4
4511	1.000	2	8423	1.000	2
4911	1.060	12	8613	1.000	2
5111	0.972	1	8741	1.130	1
5315	1.200	11	9223	1.040	1
5326	1.000	2	9523	0.910	12
5711	0.305	2	9613	1.000	2
5812	1.100	1	9713	1.250	11
5812	1.000	1	9914	1.000	2

Table B-2. Tabulation of Results for Chromium

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.120	11	4116	0.190	2
2	0.170	2	4211	0.214	2
7	0.112	5	4511	0.203	2
9	0.250	13	5111	0.038	1
12	0.174	1	5116	0.225	2
15	0.190	2	5315	0.165	11
1114	0.290	6	5326	0.160	2
1124	0.182	9	6711	0.191	2
1611	0.230	2	6911	0.200	3
1611	0.230	1	7122	0.200	2
2411	0.151	12	8112	0.265	1
2611	0.220	3	8613	0.200	2
3116	0.210	2	9223	0.250	1
3122	0.220	1	9523	0.260	12
3416	0.144	2	9914	0.190	2
		Sample 2, 0.100 mg/liter			
1	0.100	11	1611	0.130	2
2	0.060	2	1611	0.100	1
7	0.074	5	2411	0.101	12
9	0.100	13	2611	0.140	3
12	0.055	1	3116	0.090	2
15	0.106	2	3122	0.150	1
1114	0.180	6	3416	0.058	2
1124	0.095	9	4116	0.120	2

(Table B-2 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
4211	0.127	2	6911	0.110	3
4511	0.105	2	7122	0.100	2
5111	0.088	1	8112	0.125	1
5116	0.094	2	8613	0.100	2
5315	0.127	11	9223	0.115	1
5326	0.080	2	9523	0.125	12
6711	0.099	2	9914	0.090	2
		Sample 3, 0.050 mg/liter			
1	0.050	11	4116	0.060	2
2	0.030	2	4211	0.050	2
7	0.043	5	4511	0.039	2
9	0.050	13	5111	0.049	1
12	0.010	1	5116	0.045	2
15	0.054	2	5315	0.070	11
1114	0.070	6	5326	0.060	2
1124	0.050	9	6711	0.045	2
1611	0.075	2	6911	0.060	3
1621	0.045	1	7122	0.050	2
2411	0.054	12	8112	0.090	1
2611	0.080	3	8613	0.050	2
3116	0.040	2	9223	0.055	1
3122	0.080	1	9523	0.050	12
3416	0.026	2	9914	0.060	2

Table B-3. Tabulation of Results for Copper

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.060	11	3114	0.055	2
2	0.050	2	3122	0.040	1
5	0.000	5	3228	0.060	1
6	0.070	2	3316	0.075	5
7	0.073	5	3416	0.054	2
9	0.060	13	3516	0.150	1
10	0.120	1	3611	0.030	5
11	0.065	2	3711	0.051	1
12	0.130	1	4116	0.080	2
15	0.060	2	4211	0.080	1
1114	0.130	6	4511	0.050	2
1124	0.050	9	4911	0.062	12
1365	0.162	2	5111	0.051	1
1611	0.089	2	5114	0.075	2
1611	0.056	1	5315	0.052	11
1814	0.050	8	5324	0.050	2
2216	0.090	1	5711	0.050	2
2223	0.050	2	5812	0.110	1
2311	0.100	10	5817	0.060	1
2411	0.074	12	6117	0.035	2
2611	0.100	3	6711	0.038	2
2626	0.096	1	6911	0.070	3
2826	0.055	5	7122	0.006	2
3111	0.050	2	7865	0.000	2

(Table B-3 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
7866	0.200	7	9223	0.080	1
8117	0.070	1	9523	0.050	12
8423	0.054	2	9613	0.100	2
8613	0.050	2	9713	0.070	11
8741	0.050	1	9914	0.060	2
		Sample 2, 0.250 mg/liter			
1	0.280	11	2411	0.324	12
2	0.240	2	2611	0.400	3
5	0.200	5	2624	0.290	1
6	0.140	2	2924	0.252	5
7	0.465	5	3111	0.250	2
9	0.310	13	3116	0.262	2
10	0.350	1	3122	0.270	1
11	0.242	2	3226	0.250	1
12	0.315	1	3316	0.275	5
15	0.250	2	3414	0.263	2
1114	0.450	6	3516	0.410	1
1124	0.230	9	3611	0.280	5
1365	0.406	2	3711	0.376	1
1611	0.283	2	4114	0.290	2
1611	0.245	1	4211	0.274	1
1814	0.240	8	4511	0.234	2
2216	0.345	1	4911	0.227	12
2223	0.250	2	5111	0.240	1
2311	0.100	10	5114	0.225	2

(Table B-3 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
5315	0.270	11	8117	0.230	1
5326	0.250	2	8126	0.140	4
5711	0.229	2	8423	0.256	2
5812	0.300	1	8613	0.270	2
5812	0.250	1	8741	0.251	1
6117	0.235	2	9223	0.290	1
6711	0.251	2	9523	0.250	12
6911	0.270	3	9613	0.300	2
7122	0.030	2	9713	0.350	11
7865	0.250	2	9914	0.260	2
7866	0.200	7			
		Sample 3, 1.000 mg/liter			
1	1.250	11	1611	1.010	1
2	1.000	2	1611	1.010	2
5	1.000	5	1814	0.960	8
6	0.480	2	2216	1.253	1
7	1.102	5	2223	1.000	2
9	1.350	13	2311	1.000	10
10	1.310	1	2411	1.245	12
11	0.995	2	2611	1.700	3
12	1.040	1	2626	1.000	1
15	1.020	2	2826	0.973	5
1114	1.100	6	3111	0.950	2
1124	0.925	9	3116	1.100	2
1365	1.576	2	3122	0.920	1

(Table B-3 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
3226	0.990	1	6112	0.950	2
3316	1.010	5	6711	1.003	2
3416	0.990	2	6911	1.130	3
3516	1.200	1	7122	0.120	2
3611	1.070	5	7865	0.950	2
3711	1.122	1	7866	0.940	7
4116	1.010	2	8112	1.000	1
4211	1.020	1	8126	1.000	4
4511	0.957	2	8423	0.050	2
4911	0.930	12	8613	1.100	2
5111	1.000	1	8741	0.892	1
5116	0.926	2	9223	1.060	1
5315	1.230	11	9523	0.940	12
5326	0.990	2	9613	1.000	2
5711	0.682	2	9713	0.115	11
5812	1.000	1	9914	1.160	2
5812	1.045	1			

Table B-4. Tabulation of Results for Magnesium

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2	0.210	2	3711	0.210	1
6	0.070	2	4211	0.209	2
7	0.224	5	4911	0.250	12
10	0.220	1	5111	0.237	1
11	0.204	2	5315	0.206	11
12	0.195	1	5326	0.215	2
15	0.208	2	5711	0.243	2
1114	0.215	6	5812	0.213	1
1124	0.190	9	5812	0.188	1
1365	0.134	2	6112	0.210	2
1814	0.280	8	6911	0.230	3
2216	0.225	1	7122	0.200	2
2223	0.200	1	7865	0.355	2
2311	0.360	10	7866	0.200	7
2411	0.202	12	8112	0.250	1
2611	0.240	3	8126	0.175	4
2626	0.210	1	8423	0.220	2
3116	0.210	2	8613	0.210	2
3122	0.206	1	9223	0.222	1
3226	0.160	1	9523	0.300	12
3316	0.222	5	9613	0.200	2
3416	0.180	2	9713	0.170	11
3516	0.220	1	9914	0.220	2
3611	0.230	5			

(Table B-4 continued)

Sample 2, 0.100 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2	0.110	2	3711	0.122	1
6	0.270	2	4211	0.108	2
7	0.113	5	4911	0.130	12
10	0.120	1	5111	0.141	1
11	0.101	2	5315	0.148	11
12	0.083	1	5326	0.124	2
15	0.110	2	5711	0.103	2
1114	0.137	6	5812	0.114	1
1124	0.268	9	5812	0.102	1
1365	0.034	2	6112	0.120	2
1814	0.140	8	6911	0.140	3
2216	0.125	1	7122	0.100	2
2223	0.100	1	7865	0.250	2
2311	0.180	10	7866	0.100	7
2411	0.105	12	8112	0.160	1
2611	0.120	3	8126	0.067	4
2626	0.110	1	8423	0.130	2
3116	0.100	2	8613	0.100	2
3122	0.187	1	9223	0.108	1
3226	0.090	1	9523	0.120	12
3316	0.139	5	9613	0.090	2
3416	0.070	2	9713	0.025	11
3516	0.120	1	9914	0.110	2
3611	0.290	5			

(Table B-4 continued)

Sample 3, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2	0.060	2	3711	0.048	1
6	0.440	2	4211	0.054	2
7	0.050	5	4911	0.065	12
10	0.060	1	5111	0.094	1
11	0.049	2	5315	0.112	11
12	0.025	1	5326	0.080	2
15	0.053	2	5711	0.065	2
1114	0.061	6	5812	0.051	1
1124	0.263	9	6112	0.050	2
1365	0.034	2	6911	0.100	3
1814	0.080	8	7122	0.050	2
2216	0.063	1	7865	0.085	2
2223	0.050	1	7866	0.050	7
2311	0.120	10	8112	0.090	1
2411	0.054	12	8126	0.025	4
2611	0.050	3	8423	0.060	2
2626	0.050	1	8613	0.050	2
3116	0.050	2	9223	0.052	1
3122	0.057	1	9523	0.060	12
3226	0.050	1	9613	0.050	2
3316	0.043	5	9914	0.060	2
3416	0.010	2			
3516	0.060	1			
3611	0.090	5			

Table B-5. Tabulation of Results for Manganese

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.050	11	4911	0.078	12
2	0.050	2	5111	0.054	1
5	0.200	5	5114	0.055	2
7	0.087	5	5315	0.050	11
9	0.050	13	5326	0.040	2
10	0.050	1	5711	0.059	2
12	0.047	1	5812	0.070	1
15	0.066	2	6112	0.050	2
1114	0.110	6	6911	0.060	3
1124	0.085	9	7122	0.050	2
2223	0.050	2	7866	0.000	7
2311	0.050	10	8112	0.050	1
2411	0.041	12	8423	0.059	2
2611	0.040	3	8613	0.050	2
2626	0.088	1	9223	0.050	1
2926	0.043	5	9 23	0.045	12
3116	0.060	2	9713	0.050	11
3122	0.030	1			
3316	0.137	5			
3416	0.043	2			
3516	0.210	1			
3611	0.030	5			
4211	0.060	2			
4511	0.050	2			

(Table B-5 continued)

Sample 2, 0.500 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.500	11	4911	0.495	12
2	0.520	2	5111	0.502	1
5	0.400	5	5114	0.575	2
7	0.538	5	5315	0.452	11
9	0.590	13	5326	0.480	2
10	0.610	1	5711	0.424	2
12	0.476	1	5812	0.510	1
15	0.520	2	6112	0.500	2
1114	0.650	6	6911	0.160	3
1124	0.770	9	7122	0.450	2
2223	0.580	2	7866	0.500	7
2311	0.500	10	8112	0.500	1
2411	0.435	12	8124	0.200	4
2611	0.490	3	8423	0.500	2
2624	0.550	1	8613	0.490	2
2926	0.509	5	9223	0.510	1
3116	0.520	2	9523	0.450	12
3122	0.520	1	9713	0.540	11
3316	0.500	5			
3416	0.485	2			
3516	0.738	1			
3611	0.320	5			
4211	0.503	2			
4511	0.546	2			

(Table B-5 continued)

Sample 3, 0.250 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.260	11	4911	0.255	12
2	0.250	2	5111	0.257	1
5	0.200	5	5116	0.340	2
7	0.279	5	5315	0.222	11
9	0.250	13	5326	0.240	2
10	0.250	1	5711	0.226	2
12	0.210	1	5812	0.250	1
15	0.256	2	6112	0.250	2
1114	0.275	6	6911	0.100	3
1124	0.370	9	7122	0.250	2
2223	0.300	2	7866	0.250	7
2311	0.250	10	8112	0.250	1
2411	0.213	12	8126	0.150	4
2611	0.240	3	8423	0.250	2
2626	0.260	1	8613	0.250	2
2826	0.[illegible]1	5	9223	0.250	1
3116	0.2[illegible]	2	9523	0.240	12
3122	0.240	1	9713	0.310	11
3316	0.272	5			
3416	0.225	2			
3516	0.445	1			
3611	0.130	5			
4211	0.262	2			
4511	0.285	2			

Table B-6. Tabulation of Results for Silver

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.080	11	3611	0.070	5
2	0.060	2	4116	0.060	2
7	0.052	5	4211	0.050	2
9	0.050	13	4911	0.035	12
12	0.068	1	5315	0.075	11
15	0.073	2	5812	0.120	2
1124	0.057	9	6911	0.080	3
2311	0.050	10	8112	0.100	1
2611	0.060	3	8423	0.049	2
3116	0.045	2	8613	0.050	2
3316	0.094	5			

Sample 2, 0.100 mg/liter

1	0.110	11	3611	0.120	5
2	0.090	2	4116	0.110	2
7	0.081	5	4211	0.095	2
9	0.100	13	4911	0.084	12
12	0.100	1	5315	0.116	11
15	0.100	2	5812	0.160	2
1124	0.080	9	6911	0.130	3
2311	0.100	10	8112	0.140	1
2611	0.100	3	8423	0.085	2
3116	0.080	2	8613	0.090	2
3316	0.170	5			

(Table B-6 continued)

Sample 3, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.220	11	3611	0.260	5
2	0.220	2	4116	0.220	2
7	0.200	5	4211	0.211	2
9	0.210	13	4911	0.219	12
12	0.197	1	5315	0.282	11
15	0.246	2	5812	0.325	2
1124	0.205	9	6911	0.300	3
2311	0.200	10	8112	0.330	1
2611	0.220	3	8423	0.205	2
3116	0.200	2	8613	0.200	2
3316	0.340	5			

Table B-7. Tabulation of Results for Lead

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2	0.200	2	3416	0.239	2
5	0.000	5	3611	0.210	5
7	0.242	5	4116	0.210	2
9	0.220	13	5111	0.403	1
15	0.190	2	5315	0.520	11
1114	0.260	6	6711	0.115	2
1124	0.215	9	7122	0.150	2
2311	0.200	10	7865	0.100	2
2611	0.230	3	9223	0.250	1
2626	0.185	1			

Sample 2, 0.100 mg/liter

2	0.090	2	3416	0.176	2
5	0.000	5	3611	0.190	5
7	0.122	5	4116	0.120	2
9	0.170	13	5111	0.097	1
15	0.099	2	5315	0.276	11
1114	0.160	6	6711	0.053	2
1124	0.055	9	7122	0.050	2
2311	0.100	10	7865	0.000	2
2611	0.110	3	9223	0.090	1
2626	0.097	1			

(Table B-7 continued)

Sample 3, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2	0.030	2	3416	0.081	2
5	0.000	5	3611	0.160	5
7	0.070	5	4116	0.060	2
9	0.100	13	5111	0.052	1
15	0.051	2	5315	0.180	11
1114	0.100	6	6711	0.011	2
1124	0.030	9	7122	0.100	2
2311	0.050	10	7865	0.000	2
2611	0.060	3	9223	0.050	1
2626	0.053	1			

Table B-8. Tabulation of Results for Cadmium

Sample 1, 0.010 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.030	11	3122	0.030	1
5	0.000	5	3416	0.008	2
7	0.012	5	3611	0.020	5
9	0.009	13	4116	0.020	2
12	0.005	1	4911	0.022	12
15	0.011	2	5315	0.008	11
1124	0.010	9	5812	0.020	1
1611	0.017	2	5812	0.024	2
1611	0.024	1	6711	0.007	2
2311	0.010	10	6911	0.009	3
2611	0.020	3	7122	0.005	2
2626	0.005	1	9713	0.028	11
3116	0.010	2			

Sample 2, 0.000 mg/liter

1	0.070	11	2641	0.050	3
5	0.040	5	2626	0.046	1
7	0.055	5	3116	0.050	2
9	0.050	13	3122	0.070	1
12	0.049	1	3416	0.050	2
15	0.049	2	3611	0.050	5
1124	0.070	9	4116	0.060	2
1611	0.059	1	4511	0.053	2
1611	0.048	2	4911	0.060	12
2311	0.060	10	5315	0.042	11

(Table B-8 continued)

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
5812	0.048	2	7122	0.050	2
5812	0.080	1	9713	0.075	11
6711	0.028	2	6911	0.044	3
		Sample 3, 0.100 mg/liter			
1	0.130	11	3122	0.120	1
5	0.100	5	3416	0.100	2
7	0.100	5	3611	0.100	5
9	0.105	13	4116	0.110	2
12	0.130	1	4511	0.106	2
15	0.097	2	4911	0.113	12
1124	0.136	9	5315	0.083	11
1611	0.107	2	5812	0.140	1
1611	0.099	1	5812	0.092	2
2311	0.100	10	6711	0.119	2
2611	0.100	3	6911	0.088	3
2626	0.095	1	7122	0.100	2
3116	0.100	2	9713	0.125	11

Table B-9. Tabulation of Results for Iron

Sample 1, 0.100 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.100	11	4116	0.130	2
2	0.090	2	4511	0.100	2
7	0.149	5	4911	0.115	12
9	0.250	13	5111	0.136	1
11	0.063	2	5116	0.058	2
12	0.045	1	5315	0.120	11
15	0.108	2	5326	0.100	2
1114	0.240	6	5711	0.130	2
1124	0.120	9	5812	0.072	1
1814	0.240	8	5812	0.090	1
2216	0.090	1	6112	0.110	2
2223	0.050	2	6711	0.067	2
2311	0.150	10	6911	0.160	3
2411	0.100	12	7122	0.100	2
2611	0.100	3	7865	0.125	2
2826	0.088	5	8112	0.260	1
3111	0.120	2	8423	0.115	2
3116	0.100	2	8613	0.100	2
3122	0.100	1	8741	0.050	1
3226	0.100	1	9223	0.100	1
3416	0.009	2	9523	0.100	12
3516	0.370	1	9613	0.125	2
3611	0.190	5			
3711	0.137	1			

(Table B-9 continued)

Sample 2, 0.500 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.500	11	4116	0.490	2
2	0.500	2	4511	0.488	2
7	0.633	5	4911	0.445	12
9	1.100	13	5111	0.519	1
11	0.456	2	5116	0.260	2
12	0.350	1	5315	0.523	11
15	0.510	2	5326	0.500	2
1114	0.585	6	5711	0.568	2
1124	0.500	9	5812	0.480	1
1814	0.590	8	5812	0.520	1
2216	0.550	1	6112	0.550	2
2223	0.500	2	6711	0.665	2
2311	0.600	10	6911	0.520	3
2411	0.503	12	7122	0.500	2
2611	0.460	3	7865	0.475	2
2826	0.440	5	8112	0.580	1
3111	0.500	2	8423	0.505	2
3116	0.480	2	8613	0.500	2
3122	0.540	1	8741	0.500	1
3226	0.520	1	9223	0.530	1
3416	0.500	2	9523	0.520	12
3516	0.930	1	9813	0.575	2
3611	0.720	5			
3711	0.512	1			

(Table B-9 continued)

Sample 3, 0.300 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.260	11	4116	0.310	2
2	0.290	2	4511	0.250	2
7	0.404	5	4911	0.285	12
9	0.700	13	5111	0.366	1
11	0.214	2	5116	0.190	2
12	0.250	1	5315	0.314	11
15	0.305	2	5326	0.300	2
1114	0.390	6	5711	0.330	2
1124	0.325	9	5812	0.349	1
1814	0.370	8	5812	0.300	1
2216	0.340	1	6112	0.320	2
2223	0.200	2	6711	0.372	2
2311	0.300	10	6911	0.330	3
2411	0.302	12	7122	0.250	2
2611	0.310	3	7865	0.300	2
2826	0.248	5	8112	0.580	1
3111	0.240	2	8423	0.325	2
3116	0.280	2	8613	0.290	2
3122	0.320	1	8741	0.210	1
3226	0.290	1	9223	0.320	1
3416	0.290	2	9523	0.300	12
3516	0.680	1	9613	0.360	2
3611	0.370	5			
3711	0.313	1			

Table B-10. Tabulation of Results for Extracted Chromium

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
10	0.210	1	4511	0.206	2
1365	0.000	2	4911	0.298	12
2216	0.011	1	5711	0.177	2
2223	0.150	2	5812	0.210	1
2626	0.212	1	6112	0.250	2
3111	0.181	2	7866	0.500	7
3226	0.430	1	8126	0.170	4
3316	0.223	5	8741	0.050	1
3516	0.015	1	9613	0.090	2
3711	0.190	1			

Sample 2, 0.100 mg/liter

10	0.120	1	4511	0.101	2
1365	0.000	2	4911	0.128	12
2216	0.003	1	5711	0.095	2
2223	0.051	2	5812	0.120	1
2626	0.117	1	6112	0.188	2
3111	0.097	2	7866	0.300	7
3226	0.100	1	8126	0.007	4
3316	0.127	5	8741	0.090	1
3516	0.011	1	9613	0.030	2
3711	0.130	1			

(Table B-10 continued)

Sample 3, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
10	0.050	1	4511	0.052	2
1365	0.000	2	4911	0.047	12
2216	0.002	1	5711	0.046	2
2223	0.100	2	5812	0.060	1
2626	0.046	1	6112	0.156	2
3111	0.046	2	7846	0.200	7
3226	0.080	1	8126	0.005	4
3316	0.020	5	8741	0.005	1
3516	0.009	1	9613	0.025	2
3711	0.062	1			

Table B-11. Tabulation of Results for Extracted Copper

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
2626	0.050	1	8126	0.074	4

Table A-12. Tabulation of Results for Extracted Manganese

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1365	0.000	2	3711	0.025	1
1814	0.050	8	4511	0.074	2
2216	0.056	1	8741	0.075	1
3226	0.050	1	9613	0.005	2

Sample 2, 0.500 mg/liter

1365	0.000	2	3711	0.202	1
1814	0.220	8	8741	0.649	1
2216	0.040	1	9613	0.150	2
3226	0.500	1			

Sample 3, 0.250 mg/liter

1365	0.000	2	3711	0.080	1
1814	0.320	8	8741	0.300	1
2216	0.046	1	9613	0.010	2
3226	0.220	1			

Table B-13. Table of Results for Extracted Silver

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1814	0.044	8	5711	0.047	2
2216	0.024	1	6112	0.128	2
2223	0.200	2	8741	0.080	1
3122	0.085	1	9613	0.050	2
3226	0.060	1			

Sample 2, 0.100 mg/liter

1814	0.070	8	5711	0.100	2
2216	0.033	1	6112	0.195	2
2223	0.225	2	8741	0.075	1
3122	0.000	1	9613	0.020	2
3226	0.100	1			

Sample 3, 0.200 mg/liter

1814	0.096	8	5711	0.208	2
2216	0.021	1	6112	0.153	2
2223	0.348	2	8741	0.005	1
3122	0.000	1	9613	0.010	2
3226	0.210	1			

Table B-14. Table of Results for Extracted Lead

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1365	0.009	2	4511	0.189	2
1814	0.194	8	4911	0.240	12
2216	0.125	1	5711	0.159	2
2223	0.300	2	5812	0.250	1
2626	0.204	1	6911	0.180	3
3111	0.241	2	7866	0.200	7
3116	0.215	2	8112	0.270	1
3122	0.200	1	8126	0.340	4
3226	0.220	1	8613	0.230	2
3316	0.245	5	8741	0.250	1
3711	0.164	1	9613	0.125	2
4211	0.228	1	9713	0.170	11

Sample 2, 0.100 mg/liter

1365	0.006	2	4511	0.107	2
1814	0.112	8	4911	0.097	12
2216	0.100	1	5711	0.096	2
2223	0.100	2	5812	0.110	1
2626	0.063	1	6911	0.110	3
3111	0.190	2	7866	0.100	7
3116	0.105	2	8112	0.140	1
3122	0.100	1	8126	0.140	4
3226	0.110	1	8613	0.110	2
3316	0.107	5	8741	0.500	1
3711	0.125	1	9613	0.050	2
4211	0.093	1	9713	0.100	11

(Table B-14 continued)

Sample 3, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1365	0.004	2	4511	0.051	2
1814	0.062	8	4911	0.054	12
2216	0.055	1	5711	0.051	2
2223	0.050	2	5812	0.050	1
2626	0.056	1	6911	0.060	3
3111	0.099	2	7866	0.080	7
3116	0.050	2	8112	0.090	1
3122	0.050	1	8126	0.060	4
3226	0.050	1	8613	0.056	2
3316	0.067	5	8741	0.210	1
3711	0.070	1	9613	0.900	2
4211	0.055	1	9713	0.025	11

Table B-15. Table of Results for Extracted Cadmium

Sample 1, 0.010 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1365	0.001	2	4511	0.010	2
2216	0.004	1	6112	0.008	2
2223	0.020	2	8423	0.011	2
2626	0.007	1	8613	0.010	2
3226	0.010	1	8741	0.030	1
3316	0.004	5	9613	0.125	2
4211	0.015	1			

Sample 2, 0.050 mg/liter

1365	0.001	2	4511	0.045	2
2216	0.008	1	6112	0.045	2
2223	0.055	2	8423	0.054	2
2626	0.042	1	8613	0.048	2
3226	0.060	1	8741	0.050	1
3316	0.058	5	9613	0.275	2
4211	0.054	1			

Sample 3, 0.100 mg/liter

1365	0.001	2	4511	0.091	2
2216	0.006	1	6112	0.083	2
2223	0.102	2	8423	0.133	2
2626	0.090	1	8613	0.100	2
3226	0.110	1	8741	0.110	1
3316	0.131	5	9613	0.590	2
4211	0.087	1			

Table B-16. Table of Results for Extracted Iron

Sample 1, 0.100 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
10	0.100	1	3316	0.325	5
1365	0.010	2	7866	0.090	7
2626	0.078	1	9713	0.230	11

Sample 2, 0.500 mg/liter

10	0.450	1	3316	0.487	5
1365	0.015	2	7866	0.480	7
2626	0.270	1	9713	0.100	11

Sample 3, 0.300 mg/liter

10	0.250	1	3316	0.394	5
1365	0.020	2	7866	0.230	7
2626	0.230	1	9713	0.640	11

Table A-17. Tabulation of Results for Magnesium (Lanthanum Added)

Sample 1, 0.200 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
1	0.210	11	8741	0.099	1
5315	0.672	11			

Sample 2, 0.100 mg/liter

1	0.120	11	8741	0.100	1
5315	0.524	11			

Sample 3, 0.050 mg/liter

1	0.180	11	8741	0.062	1
5315	0.650	11			

Table B-18. Tabulation of Results for Manganese (Calcium Added)

Sample 1, 0.050 mg/liter

LAB. NO.	RESULTS	METHOD	LAB. NO.	RESULTS	METHOD
3711	0.035	1	5315	0.048	11

Sample 2, 0.500 mg/liter

3711	0.475	1	5315	0.796	11

Sample 3, 0.250 mg/l

3711	0.220	1	5315	0.412	11

APPENDIX C.

GLOSSARY OF STATISTICAL TERMS

A glossary of statistical terms defined as they are used in this report is presented to ensure uniform and complete understanding.

Arithmetic mean — The sum of a series of test results divided by the number of tests in the series.

Median — Halfway point in the readings when they have been arranged in order of size (the middle reading of an odd number of readings, or the average of the middle two for an even number).

Accuracy — The correctness of a measurement, or the degree of correspondence between the results and the true value (actual amount added).

Accuracy data — Measurements that relate to the difference between the average test results and the true value when the latter is known or assumed. The following measures apply:

Average deviation from true value – Average difference (when always considered as positive) between the mean determination for each laboratory and the true value.

Average percent deviation from true value – Average of the differences between a laboratory's replicate results and the true value expressed as percentages of the true value.

Mean error – The average difference with regard to sign between the test results and the true results. Also the difference between the average of a series of test results and the true results.

Relative error – The absolute value of the mean error of a series of test results as a percentage of the true result.

Precision — A measure of the reproducibility of measurements, or the degree to which the measurements correspond to one another.

Precision data — Measurements that relate to the variation among the test results themselves, i.e., the scatter or dispersion of a series of test results without assumption of any prior information. The following measures apply:

Variance – Sum of squares of deviations of the average test results from the mean of the series divided by one less than the total number of average test results.

Standard deviation – Square root of the variance.

Relative standard deviation (coefficient of variation) – Standard deviation expressed as a percentage of the mean of a series of laboratory results.

Range – Difference in magnitude between the highest and lowest laboratory mean.

Average percent deviation within laboratory – Average of the differences between the replicate results from a laboratory and the mean of their results, expressed as percentage of their mean.

Confidence limits – Limits within which the true mean of the population (the theoretically infinite number of possible replications of the analysis) will lie with a given probability.

95% Tolerance limits – Limits within which one can state with chosen confidence that 95% of the individuals of a population will lie.

APPENDIX D.
TESTS FOR NORMALITY AND REJECTION OF OUTLIERS

Test for normality

The Kolmogorov-Smirnov goodness-of-fit test was used to determine whether the observations reported could reasonably be thought to have come from a normal distribution. *

Briefly, the test involves computing the observed cumulative frequency distribution (the percent of values less than or equal to each value in the distribution) and comparing it with the theoretical normal cumulative frequency distribution. The point at which the two distributions, theoretical and observed, show greatest divergence is determined. Reference of the value of the divergence to a table of critical values for the Kolmogorov-Smirnov goodness-of-fit test indicates whether such a large divergence is likely on the basis of chance. If such a large divergence is not likely, the distribution is designated as nonnormal; otherwise the distribution is designated as normal.

Tests for rejection of outliers

1. If the distribution is designated as nonnormal, the suspected outlier (the farthest value from the mean) is rejected only if the distance between it and the mean is greater than three standard deviations; otherwise the suspected outlier is accepted.

2. If the distribution is designated as normal and the sample size is less than or equal to 30, the suspected outlier, the farthest value from the mean, is tested for rejection by a method developed by Dixon. ** Briefly, this test involves computing a ratio that compares the distance of the suspected value from its neighbors with the range of all, or most all of the observations (depending on the exact number in the distribution). Reference of the ratio to a table of critical values for test ratios for gross errors indicates whether such a large ratio is likely on the basis of chance. If the ratio is greater than or equal to the critical value, the probability that the suspected outlier is from the sample distribution is small; hence, the outlier is rejected. If the ratio is less than the critical value, the suspected outlier probably came from the sample distribution; hence, the suspected outlier is accepted.

* S. Siegel. Nonparametric Statistics for the Behavioral Sciences. McGraw-Hill Book Company, Inc. New York, New York. 1956. pp. 47-51.

** W. J. Dixon and F. J. Massey, Jr. Introduction to Statistical Analysis, Second Edition. McGraw-Hill Book Company, Inc. New York, New York. 1957. p. 276.

3. If the distribution is designated as normal, and the sample size is greater than 30, the suspected outlier is tested for rejection by a method developed by Santner.[+] This method employs the statistic $\frac{\overline{X} - X_o}{s}$, where $\overline{X}$ is the sample mean, X_o is the suspected outlier (the farthest value from the mean), and s is the sample standard deviation. This statistic is compared to a table of critical values to determine whether its value is larger than would be expected on the basis of chance. If the statistic is greater than or equal to the critical value, the suspected outlier is rejected; otherwise, the suspected outlier is accepted.

Application of tests for normality and for rejection of outliers to ARS studies

The test for normality and the subsequent test for rejection of outliers are applied to the observed data in two ways; first, to each method for a given substance at a given level of concentration, and then to a given substance at a given level of concentration regardless of method. In either case, it is first necessary to determine whether the original distribution is normal or nonnormal. If the original distribution is designated as nonnormal, method 1 is used to test for rejection of the suspected outlier farthest from the mean. If the suspected outlier is not rejected, no further tests for normality or rejection of outliers are made, and the distribution is designated as nonnormal. On the other hand, if the suspected outlier is rejected, the new distribution, which excludes the rejected observation, is then tested for normality. If the new distribution is nonnormal, the next suspected outlier is tested for rejection by method 1. This cycle of testing for normality and testing for rejection of outliers continues until a suspected value is not rejected or the test for normality designates the distribution as normal. If the distribution is designated as normal, subsequent tests for rejection of outliers made by method 2 or 3 are the same as if the original distribution had been normal. This case is discussed next.

If the original distribution is designated as normal or a new distribution that was originally nonnormal is designated as normal after the rejection of one or more outliers, and if the number of observations is not greater than 30, then method 2 is used to test for rejection the suspected outlier farthest from the mean. If the suspected outlier is not rejected, no further tests are made, and the distribution is designated as normal. If the suspected outlier is rejected, then the suspected outlier farthest from the mean of the new distribution is tested for rejection, and so on until the suspected value of a new distribution is not rejected; when this occurs, no further tests are made, and the final distribution is designated as normal. On the other hand, if the number of observations in the

[+] J. Santner. Personal communication.

original distribution is greater than 30, method 3 is used to test the suspected outlier for rejection. If the suspected outlier is not rejected, no further tests are made, and the distribution is designated as normal. If the suspected outlier is rejected, then the suspected outlier farthest from the mean of the new distribution, which excludes the rejected value, is tested for rejection. Testing for outliers continues by this method until a suspected outlier is not rejected or the number of observations is no longer greater than 30, in which case method 2 is used for testing for rejection of any remaining suspected outliers.

APPENDIX E.

COMPARISON OF METHODS FOR SIGNIFICANCE OF DIFFERENCE IN PRECISION AND ACCURACY

The methods with respect to precision and accuracy are compared in two ways. In the first case, two methods are compared at a given level of concentration with respect to precision and with respect to accuracy. The variances of the two methods are first compared by the F-test [1] to determine whether there is a significant difference in the precision of the two methods. The means of the two methods then are compared by the t-test [2] to determine whether there is a significant difference in the accuracy of the two methods. The t-test employed is based on the F-test results. These two tests of hypotheses will produce one of the following results.

Outcome 1: $s_1^2 = s_2^2$, $\overline{X}_1 = \overline{X}_2$

Outcome 2: $s_1^2 = s_2^2$, $\overline{X}_1 \neq \overline{X}_2$

Outcome 3: $s_1^2 \neq s_2^2$, $\overline{X}_1 = \overline{X}_2$

Outcome 4: $s_1^2 = s_2^2$, $\overline{X}_1 \neq \overline{X}_2$

In outcome 1, we conclude that no significant difference in precision or accuracy exists between the two methods.

In outcome 2, we conclude that there is no significant difference in precision between the two methods, but there is a significant difference in the accuracy of the two methods; specifically, the method whose mean is closer to the true value is the more accurate. In outcome 3, we conclude there is no significant difference in the accuracy of the two methods, but the method with the smaller variance is the more precise.

In outcome 4, we conclude that there is a significant difference in the precision and in the accuracy of the two methods. The method with the smaller variance is the more precise, and the method whose mean is closer to the true value is the more accurate.

In the second case, more than two methods are compared at a given level of concentration with respect to precision and accuracy. Bartlett's Test [3] is used first to test the hypothesis of equality of variances in order to compare the precision of the methods. If the precision is the same, the Analysis of Variance [4] is then used to test whether a significant difference exists in the means, in order to compare the accuracy of the

methods. If there is a significant difference in the means, Duncan's Multiple Range Test[5, 6] is used to determine by which method the means differ significantly. If the precision is not the same, then the Kruskal-Wallis One-Way Analysis of Variance by Ranks[7] is used to determine whether significant difference exists in the means, in order to compare the accuracy of the methods.

Once again, there are basically four possible outcomes after the above tests of hypotheses.

Outcome 1: all s_i^2 are equal, all $\overline{X}_i$ are equal

Outcome 2: all s_i^2 are equal, not all $\overline{X}_i$ are equal

Outcome 3: not all s_i^2 are equal, all $\overline{X}_i$ are equal

Outcome 4: not all s_i^2 are equal, not all $\overline{X}_i$ are equal

In outcome 1, we conclude there is no significant difference in the precision or the accuracy of the methods.

In outcome 2, we conclude there is no significant difference in the precision of the methods; however, at least one method does differ significantly with respect to accuracy, and Duncan's Multiple Range test tells us which methods differ. For example, in comparing four methods, we might conclude $\overline{X}_1 = \overline{X}_2$ and $\overline{X}_3 = \overline{X}_4$, but $\overline{X}_1$ and $\overline{X}_2$ differ significantly from $\overline{X}_3$ and $\overline{X}_4$; or we might conclude $\overline{X}_1 = \overline{X}_2 = \overline{X}_3$ but $\overline{X}_4$ differs significantly from $\overline{X}_1$, $\overline{X}_2$ and $\overline{X}_3$.

In outcome 3, we conclude there is no significant difference in the accuracy of the methods, but the precision is different in at least one method.

In outcome 4, we conclude that both accuracy and precision of the methods differ significantly.

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APPENDIX F.
ANALYTICAL REFERENCE SERVICE MEMBERSHIP

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Alabama State Department of Public Health, Montgomery
Alabama Water Improvement Commission, Montgomery
Arizona State Department of Health, Phoenix
Arkansas Pollution Control Commission, Little Rock
Arkansas State Department of Health, Little Rock
California Department of Water Resources, Sacramento
California State Department of Public Health, Los Angeles
California State Department of Public Health, Air and Industrial Hygiene Laboratory, Berkeley
California State Department of Public Health, Sanitation and Radiation Laboratory, Berkeley
Colorado Department of Public Health, Denver
Connecticut State Department of Health, Hartford
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Florida Department of Agriculture, Tallahassee
Florida State Board of Health, Jacksonville
Florida State Board of Health, Pensacola
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Indiana State Board of Health, Indianapolis
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Lawrence Experiment Station, Massachusetts
Louisiana State Department of Health, New Orleans
Maryland State Department of Health, Division of Biochemistry, Baltimore
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Maryland State Department of Water Resources, Annapolis
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New Hampshire Water Supply and Pollution Control Commission, Concord
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New York State Conservation Department, Ronkonkoma
New York State Conservation Department, Scottsville
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New York State Department of Health, Division of Air Resources, Albany
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York Research Corporation, Stamford, Connecticut

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ACCESSION NO.

KEY WORDS:

ABSTRACT: In this study, three samples containing between 0.005 and 5.0 mg per liter of each of nine metals – zinc, chromium, copper, magnesium, manganese, silver, lead, cadmium, and iron – were provided. Each participant was requested to do a single analysis for each of the metals in each of the three samples by the provided atomic absorption spectrophotometric method. This method, depending upon the sensitivity of the instrument (burner, tube, etc., available), gave the participant a choice of aspirating the sample directly into the flame or of chelating with ammonium pyrrolidine dithiocarbamate and extraction into methyl isobutyl ketone before aspirating.

The results obtained were evaluated in terms of whether the sensitivity of the method was sufficient to permit the measurement of the metal with a reasonable degree of

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(over)

precision and accuracy at the concentration prescribed by the drinking water standards.

In the determination of zinc, copper, magnesium, and iron, the procedure of aspirating the sample directly into the flame was found to meet the established criteria regardless of which type of burner or instrument was used. The same procedure was also found acceptable for the determination of chromium, manganese, and silver, provided a Boling burner was used. Acceptable results were obtained for lead only by chelation and extraction of the sample before aspiration. In the determination of cadmium, neither procedure was acceptable.

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